Performance Factors for Filtration of Air Using Cellulosic Fiber-based Media: A Review

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The filtration of air has attracted increasing attention during recent waves of viral infection. This review considers published literature regarding the usage of cellulose-based materials in air filtration devices, including face masks. Theoretical aspects are reviewed, leading to models that can be used to predict the relationship between structural features of air filter media and the collection efficiency for different particle size classes of airborne particulates. Collection of particles can be understood in terms of an interception mechanism, which is especially important for particles smaller than about 300 nm, and a set of deterministic mechanisms, which become important for larger particles. The effective usage of cellulosic material in air filtration requires the application of technologies including pulp refining and chemical treatments with such additives as wet-strength agents and hydrophobic sizing agents. By utilization of high levels of refining, in combination with freeze drying and related approaches, there are opportunities to achieve high levels of interception of fine particles. A bulky layer incorporating nanofibrillated cellulose can be used in combination with a coarser ply to achieve needed strength in a filter medium. Results of recent research show a wide range of development opportunities for diverse air filter devices containing cellulose.

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Contents

INTRODUCTION

Dangers resulting from airborne particulates and aerosols have captured urgent attention during the COVID-19 pandemic. Aerosol particles that contain viruses can travel through the air, spreading diseases (Tang *et al.* 2006; Nazarenko 2020). According to the US Centers for Disease Control and Prevention, the wearing of a facemask, *i.e.* air filtration, is one of the most effective and immediate ways to limit the spread of viral infections (CDC 2021). These recommendations are supported by published research (Cowling *et al.* 2010; Bin-Reza *et al.* 2012; Bunyan *et al.* 2013; Nazarenko 2020). The use of such masks has become commonplace in general society, as it has been for many years within hospital critical care settings (Arnold 1938). In addition, hazardous particulates and aerosol droplets can be removed by air filtration systems that are built into building ventilation equipment (Clausen 2004; Hyttinen *et al.* 2011; Brincat *et al.* 2016; Liu *et al.* 2017; Brochot *et al.* 2019; Nazarenko 2020).

The present article mainly focuses on the current and potential role of cellulosic fiber materials employed in air filtration. A recent review article highlighted the usage of cellulose as a component in facemasks (Garcia *et al.* 2021). Although cellulose is already very widely used as a filtering medium, it faces stiff competition from other materials, especially in higher-end applications. The present review considers various pros and cons of cellulosic fiber materials specifically in air filtration equipment. Evidence is considered from diverse studies, ranging from cotton fabric facemasks to hospital ventilation systems. Aspects of the size, structure, and chemical nature of cellulosic materials are considered with respect to what is known about the mechanisms of filtration in different air environments of interest. Because nanocellulose structures can be prepared with a very high surface area and very small pores, related research will be a particular focus of this review.

A recurrent theme of this review article will be the role of moisture relative to the effectiveness of air filters. As will be discussed, various theoretical approaches have been developed based on assumed completely dry filter media and air-borne particles. Related work has dealt with similar systems in which the solids are completely immersed in aqueous solution. But real issues encountered during evaluation of air filtration systems can span a wide range of intermediate conditions. For example, it is well known that the user’s breath can dampen a facemask from the inside, while rain can dampen it from the outside. Thus, although this review’s main focus is air filtration by cellulose-containing media, some issues related to damp or wet filter media have been intentionally included within its scope.

Desired Attributes for Filter Media

The usage of any material as filter media needs to be justified based on evidence or theories related to its performance in that role (Brown 1993). Capture efficiency, the ratio of filtered particles to particles in the incoming air, is a top priority (Abdolghader *et al.* 2018). The second requirement is that the resistance to flow needs to be as low as practical, depending on the application (Belkin 1997; Morgan-Hughes *et al.* 2001; Abdolghader *et al.* 2018). This is especially important in the case of facemasks, since a high resistance of the filter media promotes a greater leakage of air around the edges of the mask. An inverse relationship between capture efficiency and resistance to flow has been observed in many cases (Soo *et al.* 2016; Chien *et al.* 2018; Dziubak and Dziubak 2020). Figure 1 illustrates three kinds of substances that may need to be resisted by air filtration media.

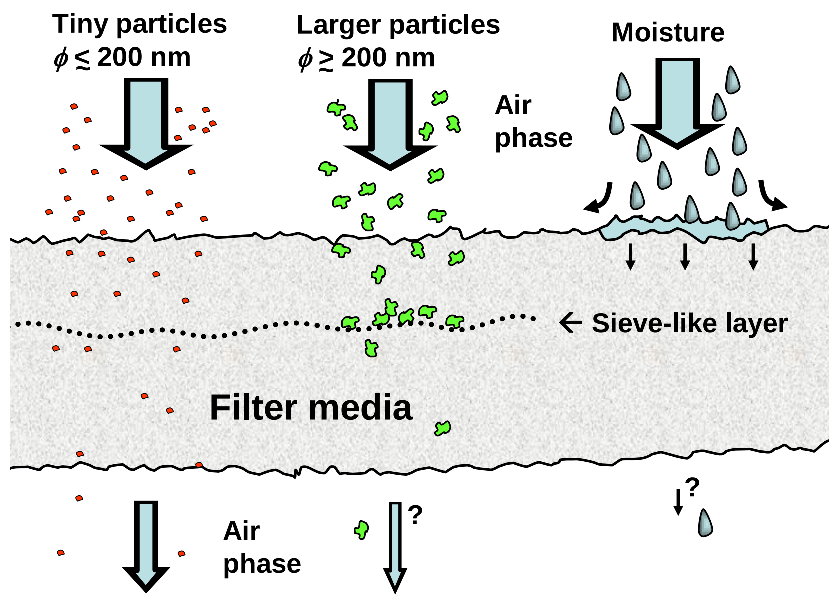

Fig. 1. Key attributes often wanted in media for air filtration, with a focus on facemasks. The horizontal dotted line represents a hypothetical deterministic, size-based filter structure that plays a role analogous to that of a sieve.

Filter media need to be tolerant of a range of moisture contents of the incoming air, including the likelihood of aerosol droplets in some cases. They need to have adequate capacity for collected particulates and not be prone to early blockage during their use. In addition, some applications can benefit from antibacterial or antiviral activities.

In common with all manufactured products, there is a preference of filter media made mostly from natural, renewable materials, using mainly eco-friendly processing conditions (Heydarifard *et al.* 2016). Cellulosic materials are often well-suited to such goals, and in addition they often have a suitably low price. Cellulose fibers, as well as various nanocellulose products, can be obtained from photosynthetically renewable plant materials. The technologies needed to separate the fibrous material, including the optional removal of lignin, are well established (Fardim and Tikka 2011). Depending on such factors as later chemical modifications, the cellulosic material is generally biodegradable (de Almeida *et al.* 2020; Li *et al.* 2021; Hu *et al.* 2022). The key questions to be considered in this article relate to how well cellulosic materials, of various types, can meet the other critical objectives and how such performance can be improved. Thus, it is important to consider the extent to which cellulose-based filter media can either match or approach the performance of filter products that have become established in the market.

Challenges for Cellulose-containing Facemasks and Filter Media

Their interactions with water represent some of the greatest differences between cellulosic materials compared to other commonly used materials for filter media. It has been found that cellulosic filtration media may be susceptible to a rise in resistance to flow after exposure to droplets of saline solution (Turnbull *et al.* 2005). The capillary forces present during ordinary drying of paper will tend to draw adjacent cellulosic surfaces into molecularly-tight contact, thus greatly decreasing the surface area available for filtration (Stone and Scallan 1966; Page 1993). Such an effect is illustrated schematically in Fig. 2. As will be discussed later, it can be a great advantage to dry cellulosic media by specialized methods such a freeze-drying in order to maintain a high specific surface area for filtration (Mao *et al.* 2008; Nemoto *et al.* 2015; Yoon *et al.* 2016; Lu *et al.* 2018; Ma *et al.* 2018; Wang *et al.* 2018). It is logical to expect that such surface area may become irrecoverably lost if the material becomes wetted and redried in the course of its usage. This type of effect will be especially important in cases where the cellulosic material has been subjected to high levels of shearing while in a wet condition, leading to a microfibrillated structure. Even ordinary cellulosic filter papers tend to have a relatively high pressure drop compared to some of the competing media, at similar levels of capture (Chien *et al.* 2018).

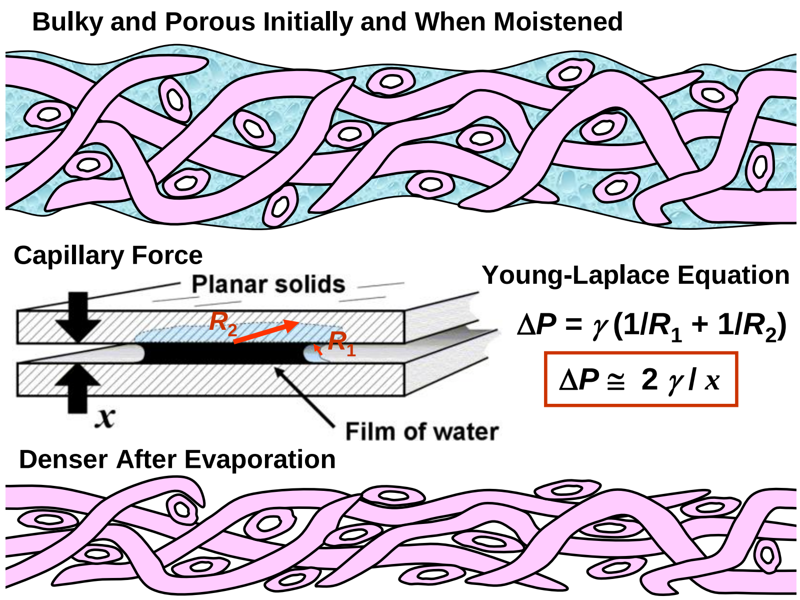

Fig. 2. Concept of how intermittent wetting and then drying may lead to densification and loss of void volume of cellulose-based filter media

Finally, there can be a concern that the hydrophilic, eco-friendly surfaces of cellulosic materials may present a favorable breeding ground for microbes. Human breath is known to be rich in microbes, including viruses (Milton *et al.* 2013). There are two parts to this concern. On the one hand, one can ask whether the hydrophilic nature of cellulose means that sufficient water to allow microbial viability will be typically present in the media during its usage. For example, cellulose-based insulation in homes can be subject to infestation by bacterial mold (Godish and Godish 2006). A study by Maus *et al.* (2001) showed that mold growth in non-cellulosic filter media was mainly dependent on the presence of nutrients and high relative humidity (> 98%). In particular, the required nutrients often can be imported by collected dust. Tests can be carried out to monitor the populations of bacteria over the course of using cellulose-based filtration media for air (Bibeau *et al.* 2000). However, there appears to be a need for research focusing specifically on whether or not the presence of cellulose within filter media affects the development of mold. On the other hand, research results suggest that the chemical and other attributes of cellulose make it suitable for various antimicrobial treatments. Table 1 lists selected studies that have focused on the development of antibacterial or antifungal treatments of filter media containing cellulose-based materials.

Table 1. Topic Areas of Some Articles Focusing on Development of Antibacterial or Antifungal Properties of Cellulose-based Air Filter Media

Topic Area	Citation
Antibacterial activity induced by surface-initiated ATRP	Tang *et al.* 2009
Review of antibacterial air filters, some using natural products	Bae & Jung 2016
Silver nanoparticles for antibacterial effects	Praveena *et al.* 2016
Nanoparticle impregnation for enhanced antibacterial effect	Jain *et al.* 2018
Nanocellulose-based filter paper for virus removal	Wu *et al.* 2019
Evaluation of regeneration methods for used facemasks	He *et al.* 2020
Diisocyanate treatment for wet strength an antibacterial effect	Zhou *et al.* 2020b
Antifungal effect of chitosan metal oxide nanoparticles, paper filter	Jain *et al.* 2022
Review of air filters for virus-containing aerosols	Mallakpour *et al.* 2022

AIR FILTRATION: THEORETICAL BACKGROUND

Overview

Air filtration by fibrous filter media has been widely studied, with both experimental and theoretical models thoroughly developed and documented. This section covers the theoretical foundations of air filtration by fibrous filter media, examining the mechanisms of filtration such as interception, filtration by size, and rebound or retention. Air permeability of the fibrous filter media is also modeled and discussed, while investigating the trade-off between capture efficiency and permeability. Respiratory mask leakage issues due to improper mask fitting are explored, as well as the relationship between mask leakage issues and permeability, with the goal of achieving optimal breathability and capture efficiency for the wearer. Filter capacity issues related to filter porosity, filter surface area, cake filtration, and filtration mechanism are discussed. Antimicrobial applications to air filtration are also covered.

In the discussions that follow, it will be shown in various cases that filtration efficiency, as well as the variations in pressure loss across media, can be affected by various parameters, such as particle size, filter media size, surface areas, pore dimensions, relative humidity, and electrical charge effects, among others. It is important to bear in mind that there are often interactive effects among two or more such parameters. Thus, there may be seeming discrepancies between the results of nominally similar studies, and there is an ongoing need to consider many aspects of the conditions under which tests are carried out.

Interception

Flow field

Filtration models can aid in better understanding the physical principles governing the performance of fibrous filters. Mathematical expressions, both theoretical and empirical, can be used in an effort to predict filter performance as a function of structure of other attributes. The most helpful models are multi-fiber models involving a flow field of aerosol particles passing around a filter fiber, while also considering the effect of neighboring filter fibers in this flow field (Lee and Liu 1980). Much of the work on modeling these flow fields amongst an array of fibers is based on the Kuwabara-Happel flow field model (Happel 1959; Kuwabara 1959). Such models are relevant to cellulose-based materials, due to their generally fibrous nature. Factors affecting the likelihood of interception of an individual particle onto a filament of filter medium can be divided into the general categories of stochastic and deterministic mechanisms (van de Ven 1989). The stochastic mechanism, to be described below, involves random diffusion of the particle. Deterministic mechanisms include stream-line-based interception, momentum-based deviations of particle paths from streamlines, and effects due to electrical charges, among others.

Diffusional interception involving Brownian motion

When considering very small particles, especially those of about 200 nm diameter or less, one can expect that if they were to exactly follow the streamlines of flow of air through the filter media, then most of them would not directly impinge onto any surfaces. Because air cannot pass through solids objects, the streamlines all will pass around any filament of filter medium in their way. Depending on the typical size of pore spaces within a filter medium, there may be a low probability that a very small particle would get stuck in an opening too small to allow its passage. Rather, in such cases the main mechanism of impaction (possibly leading to retention by the filter) will be diffusion. In other words, Brownian motion can be expected to have a dominant effect on the collection of such particles (Alonso and Alguacil 2001; Gustafsson *et al.* 2018). The general concept is illustrated in Fig. 3. Due to thermal energy, which can express itself through collisions among air molecules and particles, the momentary paths followed by such particles will be chaotic. The chaotic motions associated with diffusion will be superimposed upon the predicted motions based on the streamlines. Each entity will have an average kinetic energy of 3/2 kT, representing motion in the three dimensions of space (Hirchfelder *et al.* 1954), where k is the Boltzmann constant and T is absolute temperature. The velocity of diffusion increases with decreasing particle size. Thus, the importance of Brownian motion in bringing about impacts of particles onto filaments of the filter media will become increasingly important with decreasing particle size. Note that these predictions generally assume that colloidal-sized particles, which are thus affected by Brownian motion, are being collected on much larger filter media (such as fibrous filter media) in the absence of moisture.

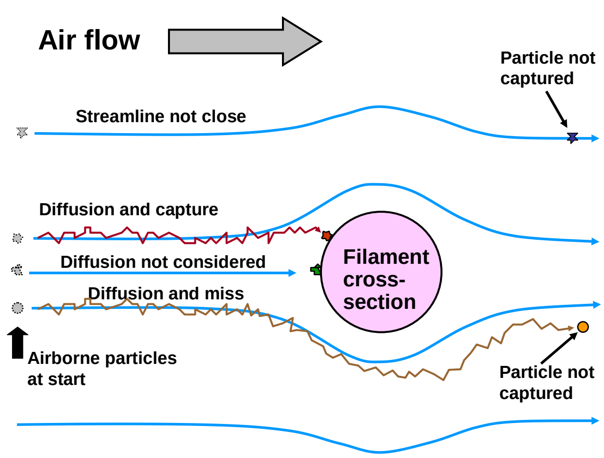

Fig. 3. General concept of a collection mechanism of very small particles that depends on their random (Brownian) movements due to their thermal energy, which is expressed by random collisions against gas molecules

Interception by the diffusion-based mechanism just described will generally increase with increasing surface area of the filter media. In the case of cellulose-based media, two aspects are critically important. Mechanical refining, hydrodynamic shearing, or micro-grinding are methods to increase the accessible surface area of cellulosic materials in the wet state (Lavoine *et al.* 2012; Gharehkhani *et al.* 2015). However, there can be a major loss in accessible surface area when cellulosic material is dried, as will be discussed later. Thus, different capture efficiencies can be expected for cellulosic filter media, in the capture of fiber small particles from air, depending on how the filter media has been prepared.

Streamline interception

Especially when the diameters of the filaments in the filter media are of similar size or smaller than those of the particles, there will be a considerable chance of direct interception, even without any need to consider effects of Brownian motion or any forces of attraction (van de Ven 1989). This type of interception is classed as deterministic, since the outcome can be predicted based on streamlines of flow. In cases where a laminar model of flow is justified, such paths can be modeled (Ayaz and Pedley 1999).

Inertial deviations from streamlines of flow

Interception of a particle *via* inertial impaction occurs when a particle's inertia causes it to stray from the original gas streamline and meet with a fiber surface (Abdolghader *et al.* 2018). This mechanism is illustrated in Fig. 4.

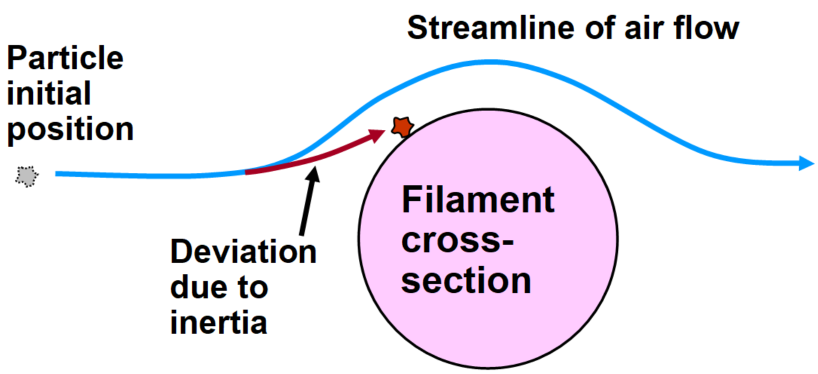

Fig. 4. Depiction of a deviation of particle motion from a streamline of flue due to the inertia of that particle, thus leading to a collision with a collector surface

The inertial filtration mechanism depends largely on the mass of the particles. The greater the particle size, the greater the inertia and the greater the inertial deposition will be. Particles with larger face velocities and densities also exhibit higher inertia.

Electrical field-induced

Filtration efficiency can be significantly improved by introduction of electrostatic forces (Abdolghader *et al.* 2018). Electrical fields can be especially useful in improving the filtration efficiency of particles that are of the wrong size to efficiently be captured by other mechanisms. This range is typically about 0.15 to 0.5 µm (see Fig. 8, with the label "Mixed capture mechanism"). However, for very small particles (smaller than 20 nm), electrical fields may decrease filtration efficiency (Zhu *et al.* 2017; Givehchi *et al.* 2015). Electrical field-induced filtration efficiency is governed by factors such as particle charge density, fiber surface charge density, the chemistry of the particles and fibers, and the intensity of the applied electric field (Mostofi *et al.* 2010).

An electrical field can be implemented either by applying a charge to the particles or by applying a charge to the filter medium (Thakur *et al.* 2013). The latter is referred to as an electret system, and an example of such a system is shown in Fig. 5. Note that such charge attraction can render the collection process much less dependent on the streamlines of flow or inertial effects. Although cellulosic materials generally are not good conductors of electricity, that does not appear to be an impediment to their usage in electret systems (Li *et al.* 2020).

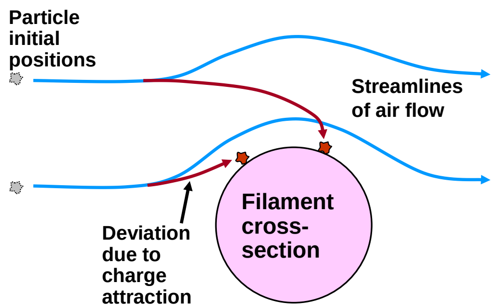

Fig. 5. Rudimentary illustration of an electret system that uses electrostatic attractive forces to enhance collection efficiency

In general, a Coulombic force is created when the charges on the particle and the fiber surface are of opposite sign. It has been stated that a polarization force is generated when the fiber surface is charged and the particle is neutral, while an image force is generated when the particle is charged and the fiber surface is neutral (Abdolghader *et al.* 2018). The word polarization is appropriate in cases where the charge distribution within a suitably small particle is capable of redistribution; in other words, sufficient electrical conductivity is a requirement. The term image force refers to a related redistribution of charge within a conductive filter surface that initially has a net neutral charge before encountering the charged particle (Muscat and Newns 1977). Such an image force has been shown to improve filtration efficiency, even for singly charged small particles (Alonso *et al.* 2007). The cited work predicts that the image force will become increasingly significant as particle size increases. In addition, the capture efficiency has been to shown to rise with increasing (opposite) electrical charge of the particles to be captured (Fjeld and Owens 1988).

Particle size is an important factor in the consideration of electrical field-induced filtration efficiency. Filtration efficiency under applied electrostatic forces has been shown to increase with increasing particle size (Thomas *et al.* 2013). The cited tests were carried out with steel or polymer fiber mesh filters having various diameters in the range of 25 to 200 μm. Larger particles have the ability to obtain more units of net charge (Hogan *et al.* 2009). Since the probability of a particle smaller than 20 nm taking on two or more net charges of either polarity is virtually zero, there are only three different conditions of charge that particles smaller than 20 nm can exhibit: neutral, singly positive, and singly negative (Hoppel and Frick 1986). Particle size is also important to consider when evaluating the charge adopted by a particle when passed through a bipolar charger. Particles smaller than 20 nm obtain different charges than larger particles when passed through the same bipolar charger and thus exhibit different filtration efficiencies. For these reasons, electrical fields applied to particles smaller than 20 nm might decrease filtration efficiency in some cases, while for larger particles, electrical field-induced filtration efficiency is reliably increased (Marlow and Brock 1975; Givehchi *et al.* 2015). The tests by Givehchi *et al.* (2015), which focused on effects due to capillary forces, involved stainless steel mesh screens with diameters of 25 μm and an air flow velocity of about 0.07 m/s.

Filtration by Size

Fiber size

Filtration of air through cellulosic fiber-based media depends partly on fiber size. Fiber diameter influences both the quality of filtration and the useful life of the filter. Fiber diameter impacts filtration characteristics such as particle penetration, most penetrating particle size (MPPS), pressure drop, and filter clogging (Chattopadhyay *et al.* 2016). The cited authors employed a commercial glass-fiber filter with a flow rate of 2.5 L/cm^2·min, which corresponds to about 0.04 m/s. The general rule is that filtration efficiency is higher for fibers with smaller diameters. Thus, it has been found that filter media composed of finer fibers exhibit a higher filtration efficiency for nanoparticles (Abdolghader *et al.* 2018). As discussed in more detail later in this article, fibrous elements in cellulose-based filter media often can be described as either "fibers" or as "nanocellulose". The diameter of a typical fiber is often in the range of 15 to 50 μm, whereas the diameter of typical nanocellulose products is often in the range of about 10 to 100 nm.

The term nanofiber has been used to describe fibers with diameters lower than 0.5 μm. Nanofibers decrease particle penetration and the MPPS of a filter, while increasing pressure drop (Kim *et al.* 2008; Chattopadhyay *et al.* 2016; Tang *et al.* 2017). High pressure drop associated with nanofibers can be attributed to nanofibers' high surface area-to-volume ratio. This characteristic renders nanofibers a poor choice for use in homogenous filter media; however, adding nanofibers as a low-density layer on top of microfibers within a composite filter achieves the benefits granted by the nanofibers while mitigating the issue of high pressure drop (Podgorski *et al.* 2006; Kim *et al.* 2008). Figure 6 presents the concept of a bulky, high surface area layer with high fractional pore volume, such that there can be a high collection efficiency of very small (Brownian-dominated) particles, while not contributing a large barrier to flow.

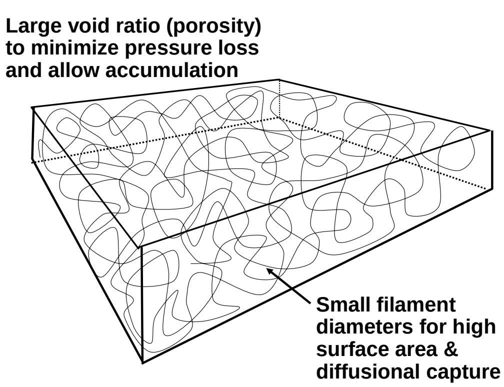

Fig. 6. Concept of a layer within a filter device that is designed to have a high efficiency of collection of particles by a stochastic Brownian diffusion mechanism while minimizing the resistance to air flow through the layer

Pore sizes in fibrous mat

Filtration of air through cellulosic fiber-based media depends partly on the pore sizes in the fibrous mat. Manipulation of pore size is therefore an important tool to improve the filtration efficiency of a filter medium. Pore size and structure of the cellulosic fiber mat significantly influences filtration efficiency due to the diffusion, interception, and sieving mechanisms (Ma *et al.* 2018). Filters with smaller pore sizes generally exhibit higher filtration efficiencies, but they also have higher pressure drops due to the smaller pore sizes (Zikova *et al.* 2015; Soo *et al.* 2016).

Another kind of deterministic capture, which is applicable to the largest dimensions of airborne particles, can be called sieving or screening. Figure 7 illustrates how this deterministic mechanism can affect particle capture efficiencies, which are expected to be a function of pore sizes.

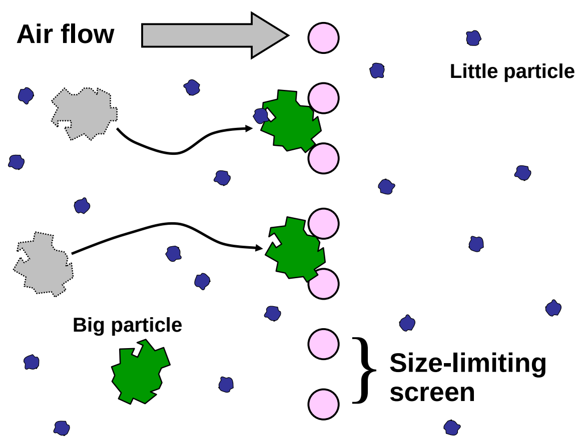

Fig. 7. Illustration a rudimentary screen-type capture, based on the sizes of particles relative to the size of passages between filaments in the filter medium

Combined effects of particle size

Based on the mechanisms already described, filtration efficiency of fibrous filters will depend in various ways upon particle size of the aerosol particles being filtered. The dominant mechanism of filtration changes depending on particle size. At the limit of very small sizes, the diffusional mechanism based on Brownian motion dominates. As particle size increases, the streamline interception and inertial mechanisms gradually become dominant. Therefore, a phenomenon exists within the intermediate particle size region in which more than one of these filtration mechanisms are operating but none is dominating. It is usually within this intermediate region that particle penetration is at a maximum and the filtration efficiency is at a minimum. Figure 8 illustrates this behavior (Lee and Liu 1980). These data were based on experiments carried out at relatively low air velocity with high-efficiency particulate air filters (HEPA).

The phenomenon of minimum filtration efficiency at a certain particle size is well established; however, the minimum filtration efficiency and the particle size at which it occurs are known to vary with the flow velocity and filter type. For fibrous filters at relatively low flow velocity, the minimum filtration efficiency generally occurs with particle diameter around 0.3 µm (Lee and Liu 1980). At higher filtration velocities, however, the most penetrating particle size may become significantly smaller in diameter than 0.3 µm (Liu and Lee 1976).

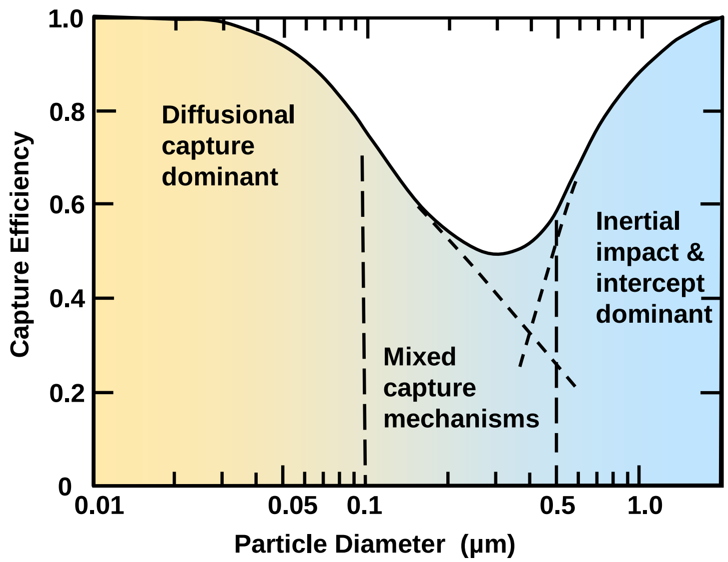

Fig. 8. Typical size-dependency of particle collection efficiency on filter media due to a transition from primarily stochastic (Brownian diffusion) capture to deterministic (direct impingement, momentum effects, *etc.*) at greater particle size. Figure redrawn based on original by Liu and Lee (1976)

Rebound or Retention

Viscoelastic properties

When a particle strikes a fiber surface within a fibrous filter under dry conditions, the initial kinetic energy of the particle is converted either to elastic deformation or the energy is lost as heat in the course of plastic deformation. In the event that all of the initial kinetic energy is consumed, the particle rests and adheres to the fiber surface. However, if the energy stored as elastic deformation exceeds the adhesion energy, then the particle will rebound from the fiber surface (Wang and Kasper 1991; Givehchi and Tan 2014). Real particles can be expected to have visco-elastic behavior, and depending on details of that behavior, different portions of the energy of impact will be irreversibly absorbed so that it no longer can contribute to the possible rebound.

A particle's adhesion to a fiber surface is partly dependent upon the particle's impact velocity. The relationship between elastic and viscous effects within a real material often can be influenced by the rate in which a process takes place. As a familiar example, very old panes of glass in windows have been predicted to be infinitesimally thicker at their bases (Gulbiten *et al.* 2018), which is consistent with a gradual process of viscous flow. But the same glass will shatter in response to a sudden impact. The fact that rebounding has been observed can be attributed to the fact that particle collection on a dry surface generally occurs during a very short time period.

When the impact velocity is less than the critical velocity, then the particles adhere to the fiber surface, whereas when the impact velocity is higher than the critical velocity, particles rebound from fiber surfaces. The particle has a greater probability of bouncing from a filter surface with increasing hardness of the contact bodies, with increasing particle size, and with increasing particle velocity (Hinds 1999). Figure 9 contrasts such bouncing with a situation in which the collector or particle is covered with a liquid, which can dissipate kinetic energy.

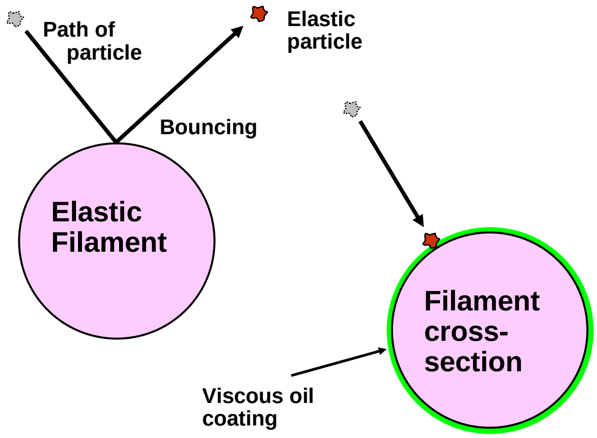

Fig. 9. Illustration of a viscous layer in determining whether an impinging particle will bounce from a collector surface or come to rest upon it

Particle shape vs. rebounding effects

With respect to particle shape, spherical particles that impinge on a collector surface may either slide or roll (Hubbe 1985; Barquins 1992). The area of contact for a spherical particle may remain nearly constant at any point in the particle's course of movement along a smooth surface. This behavior contrasts with that of cubic particles, which will either slide or tumble. As illustrated in Fig. 10, when a cubic particle tumbles along a fiber surface, the area of contact between particle and fiber changes significantly as a function of time. One can expect that the most frequent collisions will involve contact with a corner, or perhaps an edge of the cubic particle, thus involving relatively low amounts of attractive energy. By contrast, once a cubic particle comes to rest, facewise, on a flat surface, one can expect a large force of attraction; thus one can expect a correspondingly large frictional force that would resist subsequent sliding along the surface. This phenomenon leads to a greater initial probability of particle bounce in the case of cubic particles. But once the cubic particle has come to rest, presumably with flat surfaces in close contact, then it can be expected to be highly resistant to a rolling motion.

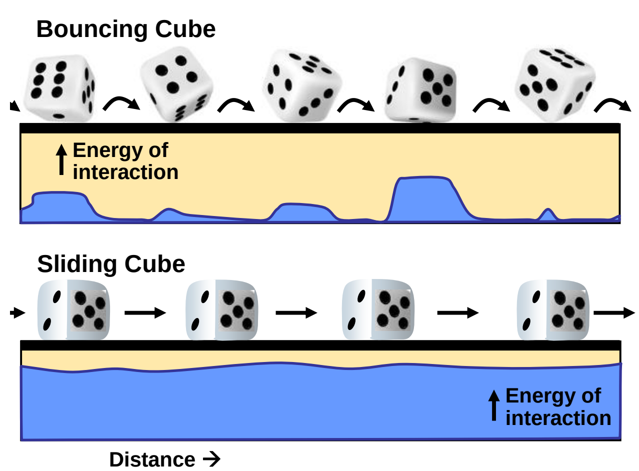

Fig. 10. Illustration of the contrasting ways in which a cubic particle would be expected to interact with a collector surface during an initial collision brought about by flow of particle-laden air through the device

Boskovic *et al.* (2005) found that, for particles between 50 and 300 nm, cubic particles experienced a lower filtration efficiency than spherical particles of the same electrical mobility diameter. This phenomenon is explained by how the different shapes physically interact with fiber surfaces (Fig. 10). The tumbling that the cubic particles exhibit can significantly alter the area of interaction between fiber surface and particle, and by this means the probability of the particle detaching from the fiber surface (particle bounce) is predicted to be high. Therefore, when all other parameters affecting filtration efficiency remain constant, the particle kinetic energy can be attributed to the difference in filtration efficiency of particles with various shapes. The higher kinetic energy of a more massive particle is demonstrated to lead to the increase in the bounce probability of a particle (Dahneke 1971; Boskovic *et al.* 2005).

Short-range attractions

Short range attractions influence the rebound or retention of a particle on a fiber surface. In particular, the London dispersion component of van der Waals forces acting between solids in an air medium will contribute an attractive component of force, regardless of the types of material. The force-distance relationship predicted for van der Waals attraction is illustrated in Fig. 11.

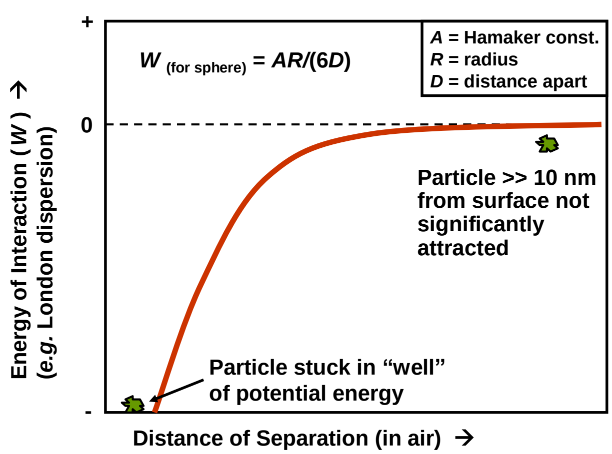

Fig. 11. Van der Waals (London dispersion component) energy as a function of distance between two solid objects

Multiple theories have been developed to calculate the adhesion energy between a particle and a surface based on elastic impaction. The most widely recognized elastic adhesion energy models are the Bradley-Hamaker (BH), Johnson-Kendall-Roberts (JKR), and Derjaguin-Muller-Toporov (DMT) models (Hertz 1882; Bradley 1932; Derjaguin *et al.* 1975; Johnson *et al.* 1971).

The Hertz elastic adhesion energy model fails to consider these short range attractions and is restricted only to small amounts of linear elasticity and deformation (Hertz 1882). Therefore, the Hertz model significantly underestimates the contact radius between particles and fiber surfaces. The BH elastic adhesion energy model does take into account van der Waals forces between two contact bodies, but it fails to consider the adhesion force from the impaction (Bradley 1932). The JKR model involves a development of the Hertz model to consider the influence of adhesion energy and contact pressure within the contact area (Johnson *et al.* 1971). Because the BH model neglects to consider specific adhesion energy between contact bodies, which plays a significant role in nanoparticle adhesion, the BH model is not useful for calculating the adhesion efficiency of nanoparticles. The JKR model, which does take this specific adhesion energy into consideration, is useful for calculating the adhesion efficiency of nanoparticles (Givehchi and Tan 2015). The DMT model includes the effect of van der Waals forces between the contact bodies (Derjaguin *et al.* 1975). The major flaw in the DMT model is that it fails to consider deformations outside the contact area (Maugis 2000). The JKR model is most applicable for soft materials, large contact radii, compliant spheres, and high adhesion energy, while the DMT model is most applicable for hard materials, small contact radii, and low adhesion energies (Maugis 2000).

Capillary Forces

Because either humidity or liquid water is likely to be present in typical situations of air filtration, significant effects of capillary forces can be expected. The capillary force effect can influence filtration performance of particles. The previously discussed adhesion energy models did not take into consideration the impact of humidity on particle adhesion energy to filter media; in these models, air was assumed to be dry. Realistically, however, ambient air usually contains moisture, so air filtration often would occur under humid conditions. These issues are especially relevant to cellulose-based filter media due to the abundant hydrophilic –OH groups at their surfaces.

Humidity in the air causes a very small meniscus to be formed in the contact area between the particle and the filter media surface (Orr *et al.* 1975; Chen and Lin 2008; Chen and Soh 2008). This meniscus expands until the condensation rate and evaporation rate reach equilibrium with the ambient air (Pakarinen *et al.* 2005). As a consequence, there is a capillary force that increases the adhesion force between particle and filter surface (Ahmadi *et al.* 2007; Zhang and Ahmadi 2007). The radius of curvature of the meniscus at equilibrium was first predicted by Kelvin (see Mitropoulos 2008).

For hydrophilic materials, the capillary force can be calculated as a function of the surface tension of water, as shown in Eq. 1,

$$F_c = 4\pi\gamma R_p (\sin\alpha \sin(\alpha + \theta) + \cos\theta) \quad (1)$$

where F_c is the capillary force, γ represents the surface tension of water (0.0735 N/m at standard temperature and pressure conditions), R_p represents the particle radius, θ represents the wetting angle, and α represents the angle between the planes perpendicular to the meniscus and filter surface. Since θ and α are usually very small, this equation can be shortened to,

$$F_c = 4\pi\gamma R_p \quad (2)$$

This equation is the standard used for spherical particles larger than about 1 µm. The physical situation is illustrated in Fig. 12. The equation may not be applicable to nanoparticles, because the capillary force for nanoparticles is partly governed by relative humidity (Pakarinen *et al.* 2005). Another reason that this equation may not be applicable to nanoparticles is that the size of the nanoparticles influences the surface tension force, which lessens the capillary force for such tiny particles (Pakarinen *et al.* 2005).

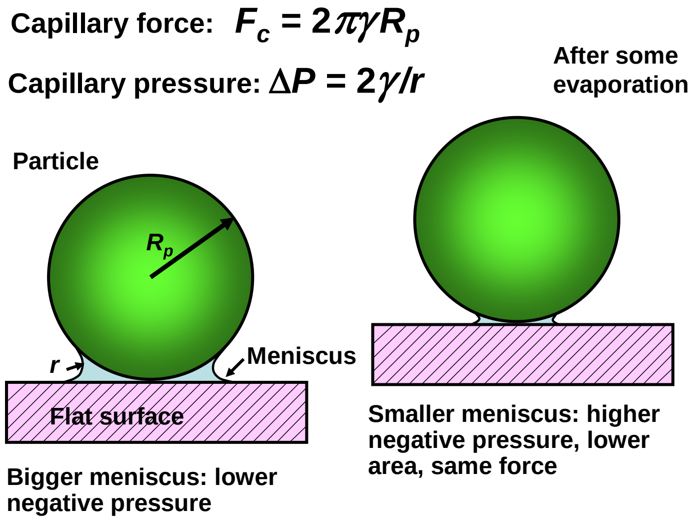

Fig. 12. Geometries for calculation of the capillary force based on the interfacial tension and the perimeter of water-air interfaces, which contribute to holding solid items together

The capillary force between nanoparticles and filter surface depends on relative humidity, particle size, and surface tension. Equations not considering capillary effects are appropriate mainly for totally dry systems or for complete immersion in liquid. To include the effect of relative humidity on capillary force, an additional term β is incorporated, which represents the ratio of the capillary force calculated using the previous equation to the actual capillary force at a particular value of relative humidity (Pakarinen *et al.* 2005). The equation for capillary force for nanoparticles to include the capillary force effect is,

$$F_c = 2\beta\pi\gamma d_p \tag{3}$$

in which β is dependent upon size and can be determined using data presented by Pakarinen *et al.* (2005). For another size and relative humidity not demonstrated in their experimental data, the capillary force can be determined by extrapolation.

Estimate of maximum negative pressure

Equations 1 through 3 were derived under an assumption that the geometry of contact between a particle and a filter surface can be well represented by a sphere interacting with a planar surface. The pressure within such a meniscus can be obtained from the Young-Laplace equation,

$$\Delta P = -\gamma \cos\theta \left[\frac{1}{R_1} + \frac{1}{R_2}\right] \tag{4}$$

where γ is the interfacial tension, θ is the contact angle of water with the surface (drawn through the water), R_1 is the smaller radius defining the meniscus, and R_2 is the larger radius defining the meniscus. The geometrical situation is sketched in Fig. 13. The greatest negative pressure can be expected when the water has a zero degree angle with the surfaces (perfect wetting); hence, the cosine term can be set equal to one. As either the meniscus continues to advance or evaporation of the water continues, the value of R_1 becomes much less than R_2, making it possible to simplify Eq. 4 as:

$$\Delta P \cong -\gamma/R_1 \tag{5}$$

At the limit where the two adjacent surfaces have come very close together, the value of ΔP is predicted to become infinitely negative. Though the validity of using the equation may become questionable at that point, what is observed in practice is that the two adjacent surfaces tend to jump into molecular contact (Campbell 1959). This mechanism helps to explain why, during the process of papermaking, it is possible to achieve high levels of relative bonded area, with the formation of hydrogen bonds directly between the two surfaces (Campbell 1959; Page 1993). Another practical consequence of such forces is that flat plates of glass can become impossible to separate if they are placed in contact while droplets of water are present.

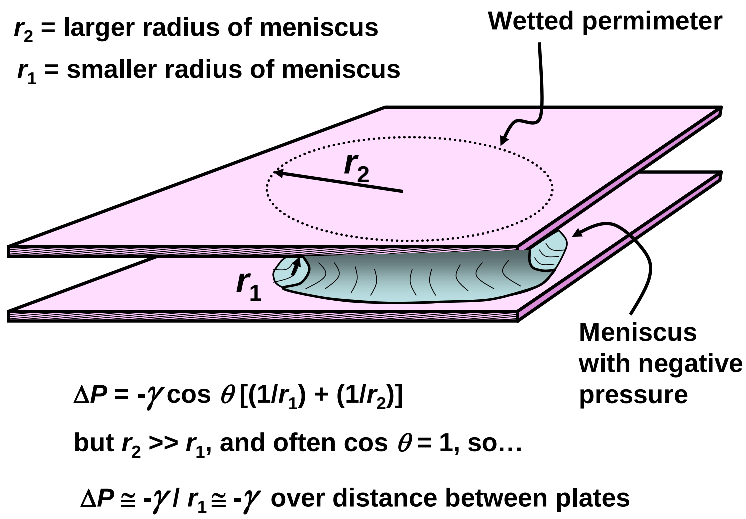

Fig. 13. Simplified view of a meniscus formed between a pair of flat surfaces what are envisioned as perfectly flat, featureless, and parallel, giving rise to a very strong negative pressure at the limit of close approach of the surfaces, based on the Young-Laplace equation

In theory, one might expect that capillary condensation would give rise to an additional component of adhesion, thus decreasing the tendency of particles to bounce away from a dry collector surface following an impingement. However, a finite time period (often measured in seconds) is generally required for the condensation to take place and for the attractive capillary force to develop (Bocquet *et al.* 1998). Since the elapsed time for rebounding of a particle will be a very small fraction of a second, there may be insufficient time for capillary condensation to have a significant effect on whether or not the particle rebounds.

Oil-coated fibers

Oil that is coated on fibers has the effect of minimizing the magnitude of particle motion along the fiber following initial particle collision with the fiber surface. This approach has been shown to be effective for air intake filters for vehicle engines (Maddineni *et al.* 2017, 2020). The level of particle motion along a filament of the filter medium will be suppressed following initial collision, which makes extensive sliding or bouncing less likely. Oil coating on fiber surfaces also raises the adhesion energy, the deformation, and the dissipative energy (Hinds 1999). The increased adhesion energy can be understood based on the simplified Young-Laplace equation (Eq. 5), where the oil is in this case playing the role of the fluid.

Boskovic *et al.* (2007) carried out an investigation in which a 3 mm-thick polypropylene medium was coated with mineral oil. The filter medium had a packing density of 0.184, and the fiber diameter averaged 12.9 µm with a standard deviation of 1.4. The particles used in this experiment were cubic MgO particles and spherical polystyrene latex particles with a diameter between 50 and 300 nm, based on electrophoretic mobility testing. Filtration efficiencies were found for two face velocities, 0.1 and 0.2 m/s. This experiment demonstrated no substantial difference in the filtration efficiencies between the spherical and cubic particles of the same electrical mobility diameter. The oil coating absorbed the particles' kinetic energy, minimizing the particle motion along the fiber following collision, and thereby reducing the probability of particle bounce. These results show that oil coating on filter fiber surfaces minimizes the effect of particle shape on filtration efficiency.

Chemical affinities

A filter demonstrates varying chemical affinities for the particulate matter or aerosols being filtered, depending on the chemical composition of the materials composing the filter. Figure 14 illustrates the concept of inter-diffusion among polymer segments at a surface, which will depend on a high level of similarity between the materials. Such similarity can be quantified based on solubility principles and involves London dispersion forces, the polar component of interaction, and hydrogen bonding ability (Hansen 2007). In principle, relatively high levels of adhesion will result in cases where the mutual solubility is high enough to allow macromolecules inter-diffusion to take place at an interface. A limitation of this mechanism of adhesion is that it requires relatively high mobility of polymer segments, *i.e.* a softened or melted condition. In addition, the passage of time is required for such inter-diffusion to take place. In the case of cellulosic filter media, especially under moist conditions of collection, one can expect strong molecular associations to form, based on solubility principles, with such materials as starches and proteins, due to chemical affinities and shared hydrophilic properties.

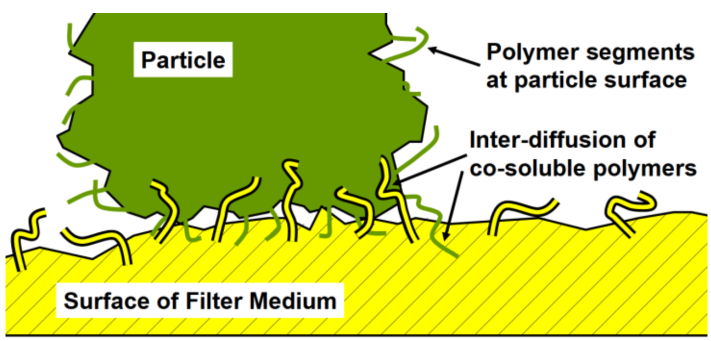

Fig. 14. The concept of inter-diffusion of polymer segments when the materials of two objects coming into contact have high similarity of such factors as dispersion interactions and polarity

Another class of chemical-based affinity is associated with triboelectric charges. Filters for medical masks and respirators are typically composed of mats of nonwoven fibrous materials, such as polypropylene, wool felt, and fiberglass paper. In electrostatic filters, resins have been implemented along with natural wool fibers to help sustain an electrostatic charge (Institute of Medicine 2006; Das and Waychal 2016). The charged character of the dry wool, *i.e.* its triboelectricity, can be attributed to the amine groups present within the protein that makes up the fiber (Shin *et al.* 2017). It has been shown that filters of natural fiber or cotton fabric exhibit less capability of sustaining a static charge compared to polyester woven fabrics due to their higher proclivity to water absorption (Konda *et al*. 2020a,b,c). Also, the addition of protein to cellulose-based filter media has been shown to improve collection efficiency (Liu *et al.* 2017; Souzandeh *et al.* 2017; Sun *et al.* 2022). Recently, much research has been directed toward more specialized materials to optimize the balance between filtration efficiency and pressure drop. These emerging materials include polymer nanofibrous membranes, carbon nanotubes, porous metal-organic frameworks, nanowire networks, silk, inorganic oxide fibrous films, chitosan, and cellulose (Liu *et al.* 2020; Ma *et al.* 2018; Chattopadhyay *et al.* 2016).

Permeability Models

Governing equations

A series of models have been developed to enable estimation of pressure drop both in the initial usage of a clean filter and progressively as particles build up within (as plugging) or on (as a cake) the filter media (Tcharkhtchi *et al.* 2021). A model can be regarded as a simplified version of reality that nevertheless may be able to suggest relationships between controlled parameters and observed parameters. Most of the expressions that have been developed to represent filter pressure drop are based on the cell models presented by Kuwabara (1959), Happel (1959), or the semi-empirical Davies equation (1953). These theoretical approaches, however, are typically only applicable to clean filters or filters that have reached equilibrium. The earlier Davies (1953) equation, which can be used to determine the pressure drop for a clean (dry) filter is,

$$\Delta P_0 = \frac{u_0 \mu_g Z}{d_f^2}[64\alpha^{1.5}(1 + 56\alpha^3)] \tag{6}$$

in which ΔP_0 represents the pressure drop, u_0 represents the gas velocity at the filter surface, μ_g represents the gas viscosity, Z is the filter thickness, d_f is the fiber diameter, α is the filter solidity or packing density, and the expression inside the brackets is an empirical correction in consideration of non-perpendicular fibers.

Davies modified his original approach to pressure drop to yield adequate results for early filtration stages (1973). In this expression, Davies (1973) substituted the terms for fiber diameter, d_f, and fiber packing density, α, with terms representing wet fiber diameter (d_{fwet}) and wet fiber packing density (α_{wet}). This modification yields the expression,

$$\Delta P = u_0 \mu_g L \frac{64\alpha_{wet}^2(1 + 56\alpha_{wet}^3)}{d_{fwet}^2},$$

where

$$\alpha_{wet} = \alpha + \frac{m_{liq}}{\Omega \rho_l L},$$

$$d_{fwet} = d_f \sqrt{1 + \frac{m_{liq}}{\Omega \rho_l Z \alpha}}, \tag{7}$$

and u_0 represents the gas velocity at the filter surface, m_{liq} represents the mass of the collected liquid, Ω represents the filtration surface area, and ρ_l represents the liquid density. The shortcoming of Davies's modified model is that it necessitates the perfect, uniform wetting of the fibers and the uniform distribution of liquid through the filter (Frising *et al.* 2005). Therefore, it is only applicable for the early stages of filtration. Thus, when considering cellulosic filters, it is likely that attributes of the filter media will tend to become less important with the passage of time during the filtration process.

Once a filter is no longer clean but has reached a "pseudo"-steady-state equilibrium, the expression developed by Liew and Conder (1985) can be used:

$$\Delta P_s = \Delta P_0 \left[1.09 \left(\alpha \frac{Z}{d_f} \right)^{-0.561} \left(\frac{U_0 \mu_l}{\sigma_{LV} cos\theta_c} \right)^{-0.477} \right] \tag{8}$$

In Eq. 8, ΔP_s represents the "pseudo"-steady state pressure drop, ΔP_0 represents the pressure drop for a clean filter, Z represents the filter thickness, U_0 represents the filtration velocity, d_f is the average fiber diameter, σ_{LV} is the liquid surface tension, θ_C is the contact angle, and μ_l is the liquid viscosity.

As illustrated in Fig. 15, the buildup of particles within and on filter media has the potential to profoundly affect both the collection efficiency and the pressure drop evaluated at a set flow rate through the device. Particles that accumulate within the filter media will eventually plug up the channels of flow. Particles that accumulate on the surface of the filter device can form a cake.

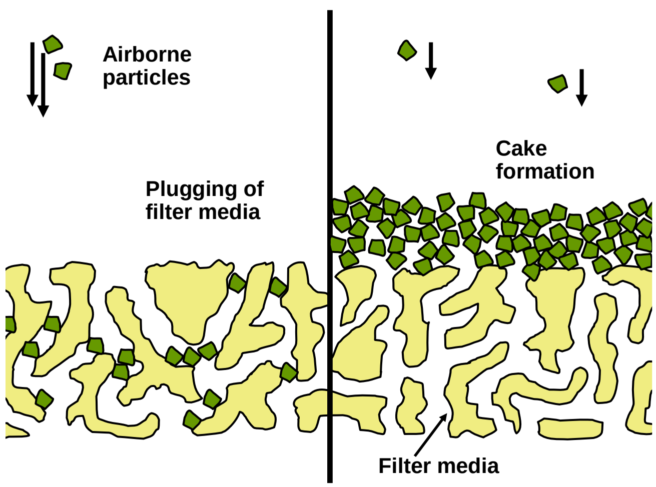

Fig. 15. Two ways in which the accumulation of particles can be effected to increase the pressure drop as particle-laden air passes through a porous filter

To account for the different stages of filtration (how factors of filtration change with filtration time), theoretical models have been developed that divide the filter into layers rather than viewing the filter as a whole. The multi-layer model proposed by Frising *et al.* (2005) includes different expressions for each of the four filtration stages proposed by Contal *et al.* (2004). In the model presented by Frising *et al.* (2005), the expression for filter penetration is,

$$P_e = exp\left(-\frac{4\alpha}{\pi d_f} dZ\right) \tag{9}$$

in which dZ represents the filter layer thickness. This model adds terms to the formula to account for the dynamic state of packing density as the filter progressively becomes clogged with more liquid. The pressure drop expressions for this multi-layer model by Frising *et al.* (2005) are derived from the 'wet' pressure drop equation presented by Davies (1973). The expression proposed by Frising *et al.* (2005) for the first stage of filtration is,

$$\Delta P = 64\mu U_0 dZ \frac{(\alpha + \alpha - l)(\alpha + \alpha_l)^{0.5}}{d_{fwet}^2}(1 + 16(\alpha + \alpha_l)^{2.5}) \tag{10}$$

in which α_l represents the liquid packing density and d_{fwet} represents wet fiber diameter. The expressions for the subsequent filtration stages are the same as this expression but with factors added in consideration of the increase in velocity through the filter that happens as it clogs. These added factors also account for the change in packing density in the filter that happens as it clogs with liquid. A source of error in this model is the theoretical assumption of a "liquid tube" model of film flow, in which Frising *et al.* (2005) theorizes a "liquid tube" forming around a fiber at the start of filtration. Mullins and Kasper (2006) demonstrated that the "liquid tube" theory is unsupported; they found that a continuous liquid film as indicated by the "liquid tube" model cannot exist in the absence of droplets, because a film in contact with an individual filter fiber will be fragmented by Plateau-Rayleigh instability (Plateau 1873). Another shortcoming of this model is the inability to predict when the transition to the next filtration stage or "layer" in the model occurs.

Blockage & the trade-off between capture and permeability

Blockage is important to consider when evaluating filtration efficiency and permeability of the filtration material. Over time, filtration efficiency increases due to clogging of the fibrous filtration media, and permeability decreases. As particle loading increases, the filtration medium's properties change and parameters influencing filtration efficiency and permeability become more complex with time (Hubbe *et al.* 2009; Mahdavi *et al.* 2015). Such an effect is illustrated in Fig. 16.

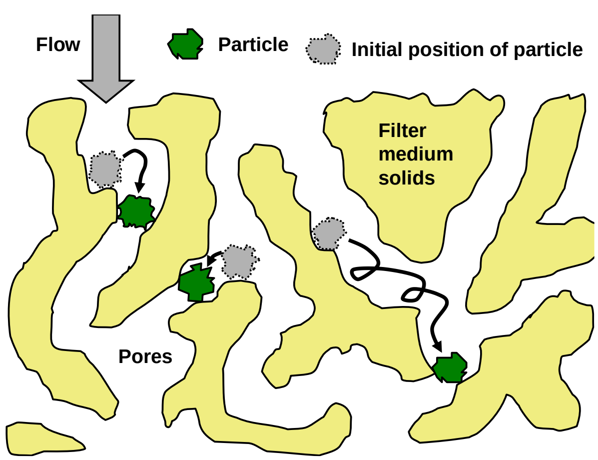

Fig. 16. Illustration of blockage of flow channels within porous media due to motions of unattached particles, which then become lodged at points of narrowing, which can result in a disproportionately adverse effect on flow through the material

A model for filtration efficiency that accounts for blockage was proposed by Hinds and Kadrichu (1997) and Kirsch (1998), in which the increase in packing density of the filter medium and the increase in fiber diameter with particle loading is considered. In the model by Hinds and Kadrichu (1997), the new packing density (α), and the new mean fiber diameter (d_f) of a clogged filter medium are represented by:

$$\alpha = \alpha_f + \alpha_p$$
$$d_f^* = \frac{d_f L_f' + d_f L_p'}{L_f' + L_p'} \tag{11}$$

In Eq. 11, L_f' signifies the fibers' total length per unit volume and L_p' represents the chain length of particles per unit volume. These terms are given by,

$$L_f' = \frac{4\alpha_f}{\pi d_f^2} \tag{12}$$

and

$$L_p' = N d_p L_T \tag{13}$$

in which N represents the number of captured particles per unit volume and L_T represents the relative length of the particle chains in regard to the fibers. This model of dynamic filtration shows that as particle loading increases, the filter medium becomes a new medium with new characteristics.

Due to its smaller size and deeper extension into the aerosol stream, a deposited particle on a filter fiber surface is generally more efficient than a fiber in collecting particles. When a deposited particle captures another particle, a dendrite may be formed that protrudes into the aerosol stream. More particle loading gives growth to more dendrites that combine in a bridge-like fashion to become a dust cake (Kanaoka *et al.* 2001; Kasper *et al.* 2009). These captured particles, especially when they form dendrites and dust cake, tend to increase filtration efficiency and decrease permeability. Such a formation of dendrites is illustrated in Fig. 17.

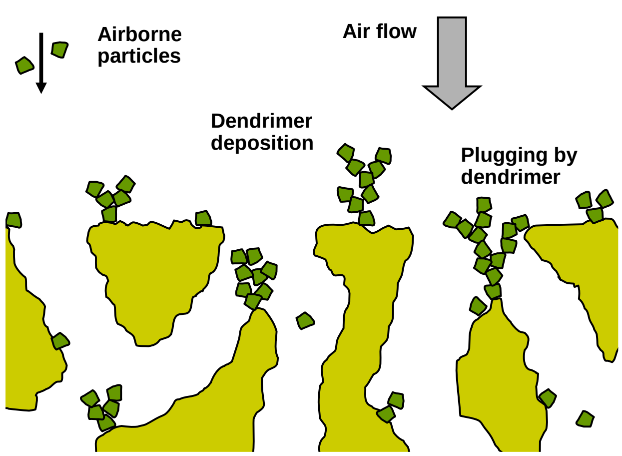

Fig. 17. Conceptual illustration of the formation of dendrites of collected particles within fiber-based filter media

The trade-off between particle capture and permeability is important to consider in the purpose of the filter media. For example, a filter in a medical mask or respirator would need to highly prioritize permeability to allow the wearer to breathe and to prevent blockage of filter pores to maintain sufficient air flow inside the filter.

Particles that are not firmly attached to surfaces within filter media can give rise to increased blockage of pores as flow continues (Hubbe *et al.* 2009). Such effects can be expected to be important for relatively deep filter beds. In principle, a detached particle will tend to follow streamlines of flow. When such a streamline encounters a sufficiently narrow passage, that is the location where the particle will get stuck, thus decreasing the permeability of the bed. Though related theory and predictions are best developed for liquid flow through packed beds and geologic beds (Davudov and Moghanloo 2019; Miri *et al.* 2021; Yang *et al.* 2022), the same principles can be expected to apply in air filtration in some cases.

Leakage Issues

Fitting

A concerning factor affecting filtration efficiency in facemasks is the fitting of the mask (Grinshpun *et al.* 2009). As illustrated in Fig. 18, due to the differing curvatures and sizes of faces from person to person and to the varying shapes of the masks, issues with sealing of the mask can arise. Leakage due to improper fitting of the mask to the wearer highly diminishes the filtration efficiency of the mask (Oberg and Brosseau 2008). Leakage can be expected especially important during exhaling, since the exhaled area will tend to push the mask away from the face. Potential ways to overcome fitting issues will be considered later, in the context of the morphology of facemasks.

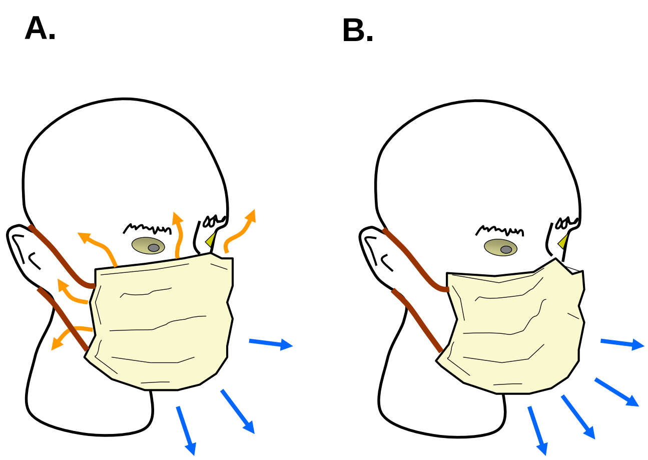

Fig. 18. The importance of achieving an excellent seal to prevent unfiltered air from bypassing the filter medium of a facemask. Figure concept redrawn based on original by Chiera *et al.* (2022)

Relationship of leakage to permeability

Permeability is a very important property to consider when constructing a practical and effective mask. Figure 19 illustrates a commonly observed trade-off between collection efficiency and pressure drop through filter media, for instance when the thickness of the filter device is increased. Respiratory resistance in a mask will inhibit the user from properly wearing a mask. However, too much permeability will cause microbes and particles to leak through the pores of the mask. N-95 masks are highly efficient in filtering viruses and bacteria, but their poor permeability can create a hypoxic environment within the mask. Huang and Huang (2007) showed such oxygen concentrations to be as low as 16.4%, which is not practical or safe for long-term wear. It has been demonstrated that using nanofibers in suitably low-density filter media increases permeability and thus decreases respiratory resistance (Skaria and Smaldone 2014). This higher permeability leads to more of the exhaled air passing through the mask filter fibers rather than the exhaled air exiting the sides of the mask and bypassing the filter fibers. As shown in Fig. 19, due to a variety of different mechanisms, the relationship between pressure drop and capture efficiency is likely to be nonlinear. Regarding leakage, a finite amount of air might bypass a filter by diffusion (probably a minor proportion), and thereafter the leakage might be directly proportional to the pressure drop across the filter device.

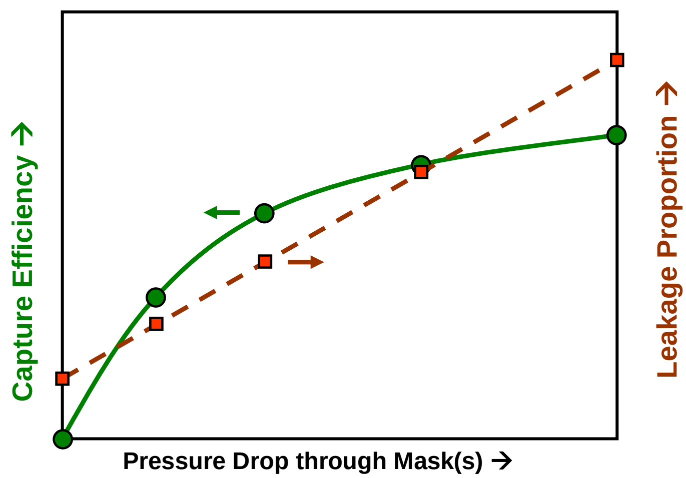

Fig. 19. Hypothetical illustration of an expected trade-off between collection efficiency and pressure drop, especially when using a simple uniform layer of filter media

Filter Capacity Issues

Pore volume fraction, media surface area, and mechanism-dependence of filter capacity

A filter medium's filter capacity is dependent, among other factors, upon both its specific surface area and its pore volume fraction. Formulating a filter with specific surface area high enough for sufficient filtration efficiency and with a pore volume fraction high enough for appropriate permeability, without sacrificing filtration efficiency, is the main challenge. From a rudimentary perspective, a filter medium's filter capacity is a function of the pore volume fraction and the surface area of the filter medium; however, filtration is a highly complex process to model, and other factors and mechanisms must be evaluated (Guo *et al.* 2002).

Figure 20 depicts two idealized views related to filter capacity and how particles can accumulate. If and when a cake of collected particles has covered the surface of a filter device, the internal capacity of the filter medium may sometimes become unimportant relative to that of the cake material (Gupta *et al.* 1993).

In addition, the holding capacity of filter media can be affected by the conditions of testing. For instance, Pei *et al.* (2019) showed that the capacity of a cellulose filter for KCl particles increased with increasing relative humidity. The cited authors explained their findings based on a capillary condensation mechanism (Mitropoulos 2008), followed by partial dissolution of the particles in the lenses of aqueous solution resulting from the condensation. Such capillary condensation, which occurs at the nano-sized points of contact between solid surfaces, explains why dry solid particles collected on a dry solid surface tend to become more firmly attached during the first few seconds or minutes of contact in the presence of ambient air (Bocquet *et al.* 1998).

A key limitation regarding capacity of filter devices is a tendency for blockage of flow, either by cake formation of debris on the surface of the filter or by clogging of pores within the media (see Fig. 20). Such blockage can render unavailable some of the initial capacity of the filter media to accommodate materials. Rochereau *et al.* (2008) considered cases in which cellulosic material was regarded as "non-adsorptive" for the material that was being collected from the air. However, the presence of cellulose appeared to increase the capacity of the filter to contain the silver aerosol particles. Particle loading also can affect the subsequent collection efficiency (Wang and Otani 2013).

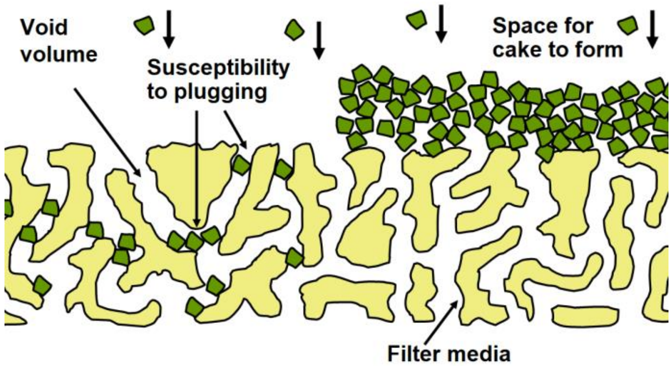

Fig. 20. Depiction of factors affecting the capacity of a filter device, including void volume, plugging (which can render some volume unavailable), and cake formation

From the standpoint of cellulosic materials preparation, some known technologies can be used that will affect the proportion of void volume and the specific surfaces area of a resulting filter mat. Three stages of such preparation need to be considered, namely the preparation of the wet fibers, the forming of the mat, and the drying. The drying stage is especially critical. Capillary forces during the conventional drying of cellulosic fiber mats have the potential to close pores and densify that material (Rey and Vandamme 2013; Akbari *et al.* 2015; Yamasaki *et al.* 2019; Ben Abdelouahab *et al.* 2021). Yamasaki *et al.* (2019) found that shrinkage of nanocellulose-containing gels during their drying could be minimized by the three strategies of (a) using inherently stiff structural components (*e.g.* the cellulose nanocrystals themselves), (b) changing the interactions between the particles, and (c) using solvent exchange as a means of reducing the capillary forces during drying. Other reported approaches to achieve the same ends include freeze drying (Mao *et al.* 2008; Jimenez-Saelices *et al.* 2017). Jimenez-Saelices *et al.* (2017) demonstrated that a combination of spray drying and freeze drying outperformed ordinary freeze drying with respect to preserving the mesopore structure of NFC aerogels. Toivonen *et al.* (2015) achieved a higher rate of production of NFC aerogel films by use of a solvent exchange procedure before drying. The water was first exchanged to isopropanol and then to octane, followed by evaporation under ambient conditions. Options of this nature will be explored in greater detail in the next section of this article.

To combat filter capacity issues and to optimize filter efficiency while maintaining a low pressure drop, the choice of filter medium is a highly important factor. Fibrous filter membranes have superior filter capacity in comparison to porous films because porous films operate mainly by the size-exclusion principle, requiring their surface pores to be small enough to efficiently filter; this low surface porosity greatly increases pressure drop when the filtered particulate adheres to the pores (Zhao *et al.* 2017). Fibrous filter membranes, by contrast, are composed of fibers whose diameters can be manipulated and that are arranged by random stacking of various fiber layers. Rather than only the size-exclusion principle, fibrous filters function by a combination of other capture mechanisms, including diffusion, interception, and impaction (Li *et al.* 2013).

To enhance filter capacity, much research has been devoted to developing filter media that optimize media surface area and pore volume fraction. This can be achieved by manipulation of fiber properties. Reducing fiber size to the nanometer scale grants the filter medium a high specific surface area, which can greatly improve filter capacity (Xu *et al.* 2016). Because of the high surface area-to-volume ratio of nanofibers, filter media composed solely of nanofibers usually cause higher pressure drops than filter media composed of larger diameter fibers (Podgorski *et al.* 2006, Kim *et al.* 2008). This issue can be alleviated by manufacturing multi-layer filter media, in which a nanofiber layer is applied atop a microfiber layer, so that the resulting composite filter inherits advantages intrinsic to both fiber types composing the filter layers (Podgorski *et al.* 2006; Wang *et al.* 2008; Leung *et al.* 2010). Promising nano-scale filter media, due to their high specific surface area and highly interconnected pore structures, include polymer nanofibrous membranes, carbon nanotubes, nanocellulose, porous metal-organic frameworks, and nanowire networks (Chattopadhyay 2016; Fan *et al.* 2018; Liu *et al.* 2020).

Cake filtration

Determining a filter medium's filter capacity is further complicated by cake filtration, which was illustrated in Fig. 20. Particles become caked onto the filter surface *via* surface filtration (Kanaoka 2019). Effects related to cake formation often become evident later in the stages of filtration after enough particles have deposited on the filter surface. Cake filtration typically decreases the filter's air permeability (Ellenbecker and Leith 1980; Cheng and Tsai 1998). During filtration through a mask, bioaerosols such as bacteria, fungi, and viruses can cake on the filter surface and clog the pores of the filter, leading to decreased mask breathability and increase in the chance of secondary contamination (Chua *et al.* 2020). These phenomena can motivate the application of antimicrobial treatments in filters. Nanocellulose, due to its capacity for functionalization and thus biocidal modification, is a suitable filter membrane material to combat the issues of particle accumulation. Likewise, nanocellulose's capacity for fibrillation can help to reduce the effects of particle accumulation on filter permeability. A fibrillated nanocellulose surface increases the surface area for particle collection, reducing the frequency of plugging and caking. Freeze-drying the fibrillated nanocellulose filter to preserve the wet fibrillated structure into the dry state also helps to reduce plugging effects and increase permeability, because this structure preservation prevents loose fibrils from falling and clogging the porous fibrous network (Mao 2008).

Modeling filter capacity becomes complicated, as cake filtration is a dynamic process that causes variable thickness as well as changes in porosity and permeability. The packing density (α_{pc}) of the filter cake is an important measurement for evaluating the pressure drop of the caked filter medium. The mass of the deposited particles is found by measuring the difference in the mass between the clean filter and the clogged filter (Abdolghader 2018). In other words, the packing density of the deposited particles can be estimated from the mass of the deposited particles (m) relative to the area of the filter surface (Ω). The packing density of the cake is therefore,

$$\alpha_{pc} = \frac{m}{\Omega \rho_p L} \tag{14}$$

in which L is medium thickness and ρ_p is the particle density (Abdolghader 2018). Cake formation typically increases filtration efficiency and decrease permeability. Due to a particle's smaller size and protrusion from the filter surface into the filtrate stream, a deposited particle generally is more efficient than a fiber in collecting particles (Kanaoka *et al.* 2001; Kasper *et al.* 2009).

Antimicrobial Effects

Antimicrobial agents and their mechanisms

Antimicrobial agents can be used in filters to help prevent biological contamination. Antimicrobials can work by either by killing the microbe or by inactivating the microbe, thereby granting the host immune system an opportunity to react (Imani *et al.* 2011). Biocides have several mechanisms of killing or inhibiting microbes. Biocides can cause cell death by generating reactive oxygen species, such as hydroxyl radicals or hydrogen peroxide, which lead to the peroxidation of the phospholipids in bacterial cells to damage bacterial DNA (Choi and Hu 2008; Ruparelia *et al.* 2008). Biocides can work by physical destruction of the cell membrane or cell wall, especially during interactions with sharp edges of nanomaterials (Akhavan and Ghaderi 2010; Parandhaman *et al.* 2015). Biocides can also release metal ions that disrupt DNA replication and ATP production, leading to cell death (Gunawan *et al.* 2011). Incorporating biocides into filters can help address biological contamination issues of filters.

Cationic polymers

Since many microbes have a net anionic charge in aqueous media, cationic polymers can be highly effective for biocidal application in filters. Masks composed of polypropylene fibers treated with dimethyl-dioctadecyl-ammonium bromide have been shown to increase mask efficacy due to the positive electrical charge of the agent attracting bacteria (Huang and Huang 2007). Chitosan, due to its natural abundance, biocidal activity, and biodegradability, is a favorable cationic polymer for biocidal filter treatment (Ye *et al.* 2005). Imani *et al.* (2011) developed an antibacterial cellulose filter paper using chitosan and silver nanoparticles deposited on the cellulose fibers. They demonstrated that the chitosan and silver nanoparticles exhibited significant antibacterial activity against *Staphylococcus aureus* and *Escherichia coli*, with the 8-bilayer treated filter paper having higher antibacterial activity than the 2- or 4-bilayer treated filter paper. They found that the deposition of the chitosan and silver nanoparticles on the cellulose fibers did not affect filter pore structure or tensile strength of the filter paper.

It is known that bacterial cells tend to spread out on surfaces, and such processes may be related to interfacial forces (Hubbe 1981). If those forces are large, as in the case of a negatively charged biological cell on a positively charged solid surface, a greater degree of spreading and stress within the cell may be expected. In principle, the induced stress on the adhering microorganisms may interfere with their normal functions. Such a mechanism was demonstrated by Tyagi *et al.* (2019), in the case of CNC and the cationic polymer chitosan coated onto cellulose.

Nano-silver particles

Nano-silver particles are commonly used in commercial products that require antimicrobial activity. The success of Imani *et al.* (2011) in developing an antibacterial cellulose filter paper by treatment with nano-silver particles and chitosan already has been discussed. Kharaghani *et al.* (2018) developed an antimicrobial mask by combining polyacrylonitrile nanofibers with highly dispersed nano-silver particles. The result was a washable mask that can prevent the two-way passage of bacteria from person to environment and from environment to person. Hiragond *et al.* (2018) incorporated nano-silver particles into a commercial face mask. They found the product to exhibit high antibacterial activity toward both *Escherichia coli* and *Staphylococcus aureus*.

Used filters as source of microbes and toxins

It is well documented that used filters can be less efficient at improving air quality than a new filter. Used filters also can be a source of microbes and toxins. The microbes themselves are not always the direct cause of the filter's deterioration of air quality; the cause of reduced performance also be the chemicals left behind by microbes even after the microbes have been sterilized from a filter (Clausen 2004). These chemicals that build up on a used filter can be toxic themselves, but they also can react with ambient chemicals to form other noxious products that deteriorate perceived air quality (Weschler and Shields 1997; Weschler 2000, 2003; Clausen 2004).

Disinfection of used filters

Disinfection of used filters is especially important in the case of the repeated wearing of masks. The reuse of masks may become needed during health crises such as the COVID-19 pandemic to ensure continued availability of PPE. Some types of chemical disinfection have been shown to be ineffective and cause harm to the filter fabric (Derraik *et al.* 2020). Disinfection treatments for masks that have been demonstrated effective and to allow the safe reuse of masks include ultraviolet germicidal irradiation, application of vaporized hydrogen peroxide, and moist heat (Derraik *et al.* 2020; Rodriguez-Martinez *et al.* 2020; Singh *et al.* 2020).

FACTORS AFFECTING FILTER PERFORMANCE

Overview

Regardless of theoretical considerations and fits to mathematical models, a great deal has been reported on how various attributes of filter media affect outcomes such as collection efficiency, capacity, and pressure differential. This section will focus on such factors and their effects.

Structural Factors

Media fiber size

Fiber fineness is one of the most important parameters to consider when selecting an efficient filter medium. The sizes of the fibers composing a filter medium influence the capture efficiency of the filter. Finer fibers increase the specific surface area per unit mass of the filter. Wood cellulose fibers have a diameter around 20 μm (Chen 2017). This relatively high diameter renders unmodified wood cellulose fibers inefficient at filtering airborne aerosols (Sun *et al.* 2018; Garcia *et al.* 2021).

A wet cellulose fiber can be fibrillated to increase its specific surface area from approximately 1 to 250 m^2/g (Garner and Kerekes 1978). This increase in surface area that fibrils introduce can aid greatly in the filtration capacity. The issue of the fibrils collapsing and rebonding upon drying can be ameliorated by freeze-drying the pulp to preserve the wet fibrillated structure after the filter is dried (Fernandes Diniz *et al.* 2004; Mao *et al.* 2008; Lu *et al.* 2018). Softwood cellulose fibers have been found to yield superior fibrillation to hardwood fibers, which is possibly because of their longer length and higher capacity for mechanical abrasion (Mao 2008). Filters utilizing nanocellulose technology have been demonstrated as effective tools for air filtration to filter particulate matter (PM2.5), submicrometer aerosol particles, and microbes (Mao *et al.* 2008; Sim and Youn 2016; Chen *et al.* 2017; Chua *et al.* 2020; Garcia *et al.* 2021; Deng et al. 2022; Sun *et al.* 2022).

Cellulose pulp fibers can be mechanically refined to obtain nanofibrillated cellulose (NFC, which is also called cellulose nanofibril) or treated *via* acid hydrolysis to obtain cellulose nanocrystals (CNCs). Nanocellulose products grant the filter membrane a high filtration capacity due to their high surface area and optional surface functionalization (Alavi 2019; Sim and Youn 2016). CNC particles can have diameters of about 4 to 10 nanometers, whereas NFC is generally somewhat thicker and much longer (Donaldson 2007). High surface area can be achieved, even without going all the way to nanocellulose, by mechanically beating and fibrillating the pulp fibers, creating fibrils with a larger surface area both on the fiber surface and within in the fiber cell wall.

Media pore size and fractional porosity

Another way to visualize the microstructure of a filter is to focus on the sizes of the pores. The pore size and fractional porosity of a cellulosic filter medium are governing factors affecting a filter's capture efficiency. Due to the micrometric diameter of native cellulose fibers, ordinary cellulose pulp produces filters with comparatively large pores, which are associated with spaces between the fibers in the mat. The relatively large pores (as well as low specific surface area) result in a filter with relatively low capture efficiency against particulate matter and airborne aerosols (Garcia *et al.* 2021). Utilizing nanocellulose technology to yield much smaller fibrillar elements produces a filter with a much smaller pore size, greater capture efficiency, higher porosity, and in some cases lower pressure drop (Sim and Youn 2016; Chua *et al.* 2020; Liu *et al.* 2021). Cellulose's ability to form nanoporous membranes grants it capability to substitute for petroleum-based fibers in the production of disposable air filters and face masks (Chattopadhyay *et al.* 2016; Chen *et al.* 2017; Lu *et al.* 2018; Garcia *et al.* 2021; Deng *et al.* 2022).

The highly intricate nanoscale pore structure produced by nanocellulose can indirectly lower a filter's capture efficiency over time, as the openings either close up or get clogged. The initial pressure drop through a cellulose-based layer of filter media can be minimized by maintaining a low-density, bulky structure, but low density often implies a relatively low mechanical strength. Incorporating CNC as reinforcing particles into the cellulose nanofiber filter membrane has been shown to preserve the mechanical integrity of the nanoporous filter structure (El Miri *et al.* 2015; Zhang *et al.* 2019; Santos *et al.* 2020). Another way to enjoy the high capture efficiency and low pressure drop granted by the nanoporous network of a nanocellulosic fiber membrane is to build a composite filter structure that includes the nanocellulose layer of high porosity with a supporting layer of higher mechanical strength. Khalil *et al.* (2012) applied a cellulose fibrous aerogel layer to a glass fiber-based particulate air filter to successfully improve the filtration efficiency without sacrificing pressure drop.

Morphological issues relative to mask fitting

Morphological issues for filters are most prominent in face masks. Regardless of how high a mask's filtration efficiency is, fitting of the mask to the face and leakage remain challenges. Due to nanocellulose's high capacity for functionalization, a mask composed of nanocellulose fibers could minimize leakage issues by edging the cellulosic mask with another fiber layer that has been demonstrated to have a high capture efficiency, such as fluffed polypropylene fibers (Huang and Huang 2007). These authors found this method to have higher filtration efficiency and lower leakage in comparison to standard surgical masks and N-95 respirators.

Another method of minimizing leakage due to fitting issues is 3D printing of face masks. 3D printing of masks has been reported as an excellent manufacturing strategy to produce optimally fitting masks (Ishack and Lipner 2020; Swennen *et al.* 2020). 3D printing can even manufacture masks that first scan a person's face to match the contours of the mask almost perfectly, thus minimizing leakage (Cai *et al.* 2018; Swennen *et al.* 2020). It has been shown that 3D printing can utilize cellulose as the base material to print a custom-fit mask that is biocompatible, biodegradable, and minimizes leakage (Oladapo *et al.* 2021). In addition, a carboxymethylcellulose composite was demonstrated to be an excellent polymer for 4D printing of structures, where the fourth dimension is time. Such 4D printing technology can achieve shape memory properties even in response to environmental pressures. This type of approach would be highly desired in a reusable, properly fitting face mask.

Dynamic Factors

Pressure differential

Pressure differential is an important parameter to consider in air filtration, especially for face masks. A face mask needs to maintain a low pressure differential to enhance breathability for the wearer (Osman 2020). However, the trade-off between pressure differential and filtration efficiency presents challenges for manufacturing the ideal face mask. Typically, the larger the pores and the higher the porosity of the filter media, the lower the pressure differential, but this comes at the expense of a lessened capture efficiency. Therefore, when pursuing comfortability and practicality in the manufacturing of a face mask, the capture efficiency can suffer as the manufacturer attempts to maximize breathability.

Nanocellulose technology can help optimize the balance between pressure differential and capture efficiency in a face mask. Pressure differential is reduced with lower apparent density of the filter media, smaller pore diameter, and increased mat thickness (Huang *et al.* 2013; Shokri *et al.* 2015). Cellulose nanofibers are unique in that they contribute a high specific surface area (up to 101.8 m^2/g) and are able to compose a thin filter medium with a low apparent density, high porosity, high capture efficiency, and high breathability due to a low pressure differential (Jiang and Hsieh 2015; Sim and Youn 2016; Chua *et al.* 2020; Liu *et al.* 2021). A cellulose fibrous aerogel layer has been applied to a glass fiber-based particulate air filter while maintaining low pressure differential and high capture efficiency (Khalil *et al.* 2012). Mao *et al*. (2008) demonstrated a method of freeze-drying pulp so that the fibrillated structure in the wet state is preserved into the dry state, thus enabling the fibrils to remain anchored to fibers without collapsing and forming blockages in the fibrous network. Such densification would increase pressure differential and lessen breathability. Nanocellulose's high capacity for functionalization also aids in minimizing pressure differential. Chen *et al.* (2017) carried out hydrophobic modification of a thin, porous cellulose nanofibrous screen to successfully filter PM2.5 while maintaining a low pressure differential.

Flow velocity

Flow velocity greatly influences the performance of fibrous filters. At low flow velocity, diffusion and electrostatic forces tend to be the more dominant mechanisms at play for particle capture. At higher flow velocity, interception becomes the dominant mechanism (Mahdavi 2013), except for the smallest particles. Richardson *et al.* (2006) used various particles and manipulated the flow rate with N95 masks. It was shown that increasing flow rate increased particle penetration through the face mask filter. Konda *et al.* (2020a,b,c) also demonstrated that increasing the flow rate decreased the filtration efficiency and increased particle penetration through filters. This phenomenon is an important consideration in the construction of an effective cellulosic face mask, due to wearers' varying breathing flow rates, especially in conditions of manual labor or physical exercise. Experiments with cellulosic filter media for engine air filters have shown that high aerosol flow velocities can cause increased particle penetration and particle re-entrainment (Jaroszczyk *et al.* 1993). Particle re-entrainment in a face mask would especially be a concern for the wearer if the face mask contained no antimicrobials to kill the re-entrained pathogenic microbes. The influence of flow velocity on aerosol penetration through face mask and respirator filters has been thoroughly studied (Silverman *et al.* 1951; Hinds and Kraske 1987; Chen *et al.* 1992; Wallart 1997; Backman 1999; Berndtsson 1999), and the complexity of the variables at hand highlight the importance of incorporating biocides into masks designed to filter pathogenic microbes.

Moisture-related Effects

Relative humidity

Humidity is another parameter that affects the capture efficiency of cellulosic filter media (Mahdavi *et al.* 2015; Mostofi *et al.* 2010). As has been noted, capillary forces between the filter fiber surface and the filtrate particle have been shown to increase with increasing relative humidity, resulting in diminished rebound of nanoparticles from a filter surface. Because of this phenomenon, thermal rebound of nanoparticles is mainly possible at conditions of low relative humidity (Givehchi and Tan 2015). Another way that relative humidity affects the capture efficiency of fibrous filter media is by the moisture's influence on hygroscopic aerosol particles. The aerodynamic diameter of hygroscopic particles is influenced by relative humidity (Tang *et al.* 1997). The hygroscopic growth factor, which is the change of particle diameter in humid conditions versus dry conditions, depends both on particle size and on relative humidity. The growth factor has been shown to increase with decreasing particle size and with increasing relative humidity (Hu *et al.* 2010). The uncertainties of hygroscopic growth predictions are maximized with nanometer-sized particles. Biskos *et al.* (2006) found that at a fixed relative humidity, the growth factor of nanosized NaCl particles decreased with decreasing particle size below 40 nm.

These physical changes in particle sizes in response to varying relative humidity at the filter surface affect the filtration efficiency. It has been shown that increasing relative humidity irreversibly decreases filtration efficiency of fibrous filter media. Montgomery *et al.* (2015) showed that after NaCl and Al_2O_3 particles were loaded onto a fibrous filter at high relative humidity and subsequently exposed to clean air in dry conditions, the filtration efficiency of dust particles was significantly reduced. This lower filtration efficiency often can be attributed to the physical change in particle structure of the captured dust (Montgomery *et al.* 2015). Kim *et al.* (2006a) found relative humidity to have no effect on the filtration efficiency for particles smaller than 100 nm. Other studies have found filtration efficiency to increase with relative humidity for coarse particles (Hinds 1999; Miguel 2003).

The filtration efficiency of electret filters suffer with higher relative humidity, due to moisture's lessening the charge of the fiber and particle surfaces (Givehchi and Tan 2015). Another type of filter that suffers lower filtration efficiency with higher relative humidity is a face mask, which is concerning due to the moist conditions that occur with the wearer's breathing inside of a face mask (Li *et al.* 2006; Zhou *et al.* 2020a). As parts of the filter medium become filled with aqueous fluid, the air streamlines may be restricted to less numerous contiguous passages through the filter.

Wetted surfaces, swelling, and clumping of the media

Wetting of surfaces of fibrous filter media can harm the filtration efficiency of the filter. This makes face masks especially an area of concern due to the moist conditions that can develop as the wearer breathes inside of the face mask and as droplets are emitted while speaking (Li *et al.* 2006; Zhou *et al.* 2020a). Practical and theoretical aspects of the filtering of aerosol droplets have been reviewed by Rengasamy *et al.* (2004) and Mead-Hunter *et al.* (2014). The wetted surfaces from human breathing inside of a mask also reduce the electrostatic surface charge of the mask filter, resulting in a reduced filtration efficiency (Choi *et al.* 2021). Studies with N95 masks show that filtration efficiency is reduced in wet conditions versus dry conditions at various flow rates (Richardson *et al.* 2006). These humid conditions also produce an environment inside the mask favorable to microbial growth, which is a challenge for masks designed to filter microbes (Zhou *et al.* 2020a).

Cellulosic filters are a concern due to cellulose's intrinsic hydrophilic properties. Because of cellulose's hydrophilic properties, the cellulose fibers in a cellulosic filter naturally absorb water, which deteriorates the fiber structure, reduces the filter integrity, lowers water resistance, and lowers filtration efficiency due to fiber swelling (Mukhopadhyay 2014; Stanislas *et al.* 2021a,b). In addition to fiber swelling, a nanocellulosic mask's nanostructure is also altered by clumping of the wetted cellulose fibrils (Sjöstedt *et al.* 2015). It has been stated that the clumping of wetted cellulose fibrils can be combated is to introduce electrostatic charge onto the fiber surface to encourage the repulsion of the fibrils into individual fibrils (Sjöstedt *et al.* 2015). To prevent wetting, swelling, and clumping of the cellulosic filter structure, a cellulose-based mask must undergo treatment that grants it hydrophobic properties. Treatment imparting antimicrobial properties is also important in a cellulose-based mask to prevent humid conditions inside the mask from promoting microbial growth. Hydrogels prepared from cross-linking nanofibrillated cellulose, polyvinyl alcohol, and borax have shown excellent material stability, water repellency, and swelling retardancy (Spoljaric *et al.* 2014). This type of technology could be incorporated in the development of an effective cellulosic face mask.

Changes in stiffness of media

Cellulosic media change in stiffness is response to changes from humid conditions to dry conditions. It follows that a cellulosic filter's filtration efficiency would be affected due to changes in moisture. As can be seen with a cellulose sponge, a cellulosic filter contracts and stiffens upon drying and swells and softens upon wetting (Rey and Vandamme 2013). Stiffening of the cellulosic media upon drying can be attributed to closing of pores driven by capillary pressure (Campbell 1959; Page 1993; Rey and Vandamme 2013). Freeze-drying during the manufacturing of the cellulosic filter can help to avoid the complications of cellulosic filters stiffening and lowering filtration efficiency upon drying. Freeze-drying helps retain the cellulose fiber structure from the wet state into the dry state (Chatterjee and Makoui 1984). In freeze-drying, water transitions directly from ice state to vapor state, bypassing the capillary pressure of liquid menisci that would otherwise cause fibril rebonding and stiffening of the media and the formation of hydrogen bonds at the junctions between the previous cellulosic surfaces. It has been found that even partial freeze-drying of cellulosic air filters, in which most of the surface water is extracted by freeze drying and the remaining is evaporated by air drying, retains the cellulosic fiber structure into the dry state also with the fibrillation intact. The resulting filtration efficiency of this freeze-dried cellulosic air filter was close to the filtration efficiency of N95 commercial respirator filters (Mao *et al.* 2008).

Hydrophobic surfaces

Hydrophobic surfaces in cellulosic filter media are necessary to maintain high filtration efficiency after moistening. Top priorities are to avoid the clumping and loss of specific surfaces area, as has been discussed in earlier sections. Because cellulose is naturally hydrophilic, hydrophobic modification of the cellulose filter membrane is vital (Heydarifard *et al.* 2016; Fan *et al.* 2017; Liu *et al.* 2021). Cellulose's abundance of hydroxyl groups renders it highly capable of this hydrophobic modification (Alavi 2019). In addition to chemical resistance and water resistance, hydrophobic modification of cellulosic filter membranes enables more water vapor channels to be maintained in the presence of moisture (Liu *et al.* 2021). Silane compounds such as methyltrimethoxysilane and hexadecyltrimethoxysilane have been shown to be effective in derivatizing cellulose nanofiber filter membranes to improve the filtration efficiency, water resistance, and mechanical integrity of the cellulosic filter membrane (Liu *et al.* 2021; Ukkola *et al.* 2021). Heydarifard *et al.* (2016) prepared a hydrophobic, high tensile strength cellulosic filter for efficient aerosol particulate entrapment by crosslinking polyvinyl acetate to the cellulose fibers. Another method of imparting hydrophobicity to a cellulosic filter membrane is application of hydrophobic coatings, such as waxes, silicones, and fluorocarbon resins (Mukhopadhyay 2014). Further methods to impart hydrophobic character to cellulosic surfaces have been reviewed recently by Szlek *et al.* (2022).

Electrical Charge Effects

Electret systems

Electrostatic charge is a major governing factor contributing to the capture efficiency of a filter (Wang 2001). The presence of an external electric field, the electric charge of the collector surface, and the charge of the aerosol or particulate all help determine the filtration efficiency of a filter system. Application of electrostatic charge to a filter membrane allows the filter to collect small particles by adsorption due to their electrostatic charge. Without the applied charge, some of these small particles would penetrate the filter because only the physical sieving and diffusional impaction mechanisms of filtration would be at play, neither of which may be highly efficient at intermediate particle sizes. The filtration capacity of filters without electrostatic treatment has been found to only reach up to 85% in some cases (Iwata *et al.* 2016). Application of electrostatic charge to the filter material renders the filter surface positively charged. The negative charge of the microbes, aerosol particles, and other particulates results in their capture on the filter fiber surfaces (Winski *et al.* 2019). Electrostatic air filters have been demonstrated to maintain high filtration efficiency and low pressure drop even with changing membrane thickness (Chua *et al*. 2020). Other advantages achieved by electret systems of filtration are enhanced submicron particulate collection efficiency without increasing filter mass or density, enhanced filtration performance uncompromised by relative humidity and storage at high temperatures, and enhanced filtration efficiency without compromising air permeability (Mukhopadhyay 2014; Zhao *et al.* 2020). Face masks that incorporate electrostatic treatment can have higher filtration efficiencies than N95 masks (Konda *et al*. 2020a,b,c).

Triboelectricity

One way that electrostatic charge is applied to filter media is by triboelectrification. Triboelectricity occurs when surfaces gain an electric charge *via* frictional contact between the surfaces, which typically requires a polarity difference between two materials. For example, such effects can be achieved by the use of blends of contrasting fibers that encounter rubbing action (Drouin 2000). This required polarity difference restricts the candidacy of materials for triboelectric contact pairs. To combat this challenge, desired triboelectric properties can be engineered by chemical modification of filtration materials. For example, a needle-punched triboelectric air filter composed of polytetrafluoroethylene fibers modified by polyphenylene sulfide fibers and silica nanoparticles exhibited notably high filtration efficiency, and it showed excellent charge regeneration performance (Wang *et al.* 2019), which would be a property extremely useful for reusable face masks. The engineering of polymers to exhibit triboelectric properties also has been accomplished *via* atomic-level chemical modifications using amines and halogens (Shin *et al*. 2017). Krucinska (2002) studied the effects of various technological parameters and the performance of needled nonwoven fabrics for electret filtration.

Cellulose has potential for chemical modification to attain triboelectric properties due to cellulose's abundance of –OH groups at the surface (Tavakolian *et al.* 2020). In principle, untreated cellulosic material can be expected to acquire a negative triboelectric charge when it is rubbed against substances that readily give up an electron. Cellulose fibers are commonly used in the manufacturing of cellulose-based triboelectric nanogenerators (Zhang *et al.* 2021; Zhou *et al*. 2022), so this same technology can be applied to cellulosic triboelectric air filters. Using cellulose-based triboelectric nanogenerator technology, a face mask was constructed that effectively filtered PM1.0, PM 0.5, and PM0.3 with high filtration efficiency of 98.4%, 97.3%, and 95.0%, while maintaining a low pressure drop of 86.0 Pa. This face mask contained a self-powered cellulosic triboelectric air filter that was driven by the wearer's respiration (Fu *et al.* 2022).

Ionic charge effects due to chemical modification

Incorporating ionic charge into filter media can enhance the capture efficiency of the filter. For example, a reaction with cellulose's many hydroxyl groups can be used to prepare cationic functional groups (Alavi 2019). For a face mask with a nanocellulosic filter, cationization allows a moistened mask to attain permanent ionic charge, higher capture efficiency, hydrophobicity, and antimicrobial properties (Choi *et al.* 2021; Liu *et al.* 2021). Polypropylene fibers treated with dimethyldioctadecylammonium bromide were found to contribute a positive ionic charge that attracted bacteria to the medical mask filter, allowing the mask to attain a bacterial and viral filtration efficiency of nearly 100% (Huang and Huang 2007). A polycation, polyethylenimine, when adsorbed onto thin cellulose films, demonstrated 15-fold higher viral capture efficiency than the untreated cellulose surface (Tiliket *et al.* 2016). This finding explains the significant increase (to 99.999%) in capture efficiency of viral droplets when a medical mask's cellulose filter layer was modified to include two PEI-functionalized cellulose filters (Tiliket *et al.* 2011). The polycation on cellulose filter fibers was found to enhance capture of negatively charged microbes.

Electrostatically active nanoparticles

Another way to impart electrostatic charge to cellulosic filters to enhance capture efficiency is by incorporation of electrostatically active nanoparticles. Cellulose's abundance of polar hydroxyl groups within its structure grants it the excellent ability to bind electrostatically active nanoparticles (Shankar *et al.* 2018). The resulting composite structure of cellulosic filter fibers and nanoparticles has been shown to exhibit high porosity and large surface area, which can greatly increase filtration efficiency (Liu *et al.* 2008; Pradhan and Parida 2011). Metal and metal oxide nanoparticles exhibit antimicrobial activity due to their nano size and high specific surface area, which is useful in face masks to kill viruses, bacteria, or fungi that are collected by the mask (Zhou *et al.* 2020a). Common metal-based nanoparticles that have demonstrated antiviral activity are copper, silver, zinc, gold, and titanium (Galdiero *et al.* 2011). TiO_2 nanoparticles have demonstrated outstanding antiviral activity; in a sample of TiO_2 nanoparticles and Newcastle disease virus, its antiviral effect was attributed to its disintegration of the viral lipid membrane and to its blockage of viral attachment (Akhtar *et al.* 2019). Negatively charged ZnO nanoparticles have shown antiviral activity against herpes viruses (Mishra *et al.* 2011; Tavakoli *et al.* 2018; Cai *et al.* 2019). Copper oxide nanoparticles' inclusion in N95 masks demonstrated an uncompromised filtration efficiency with the advantage of granting the mask antiviral activity against virions that adhered to the mask.

Factors Affecting Permeability

Structural factors: Fiber diameter

Fiber diameter heavily influences the permeability of filter media. Permeability is especially important to consider in masks, which should exhibit a low pressure drop (*i.e.*, high air permeability) to enable comfortable breathing for the wearer (Osman 2020). Nanofiber filter media have been shown to increase air permeability, as long as factors such as apparent density are optimized. For masks, this higher air permeability means that more of the wearer's exhaled air passes through the mask rather than bypassing the filter and going around the mask (Skaria and Smaldone 2014). Nanocellulosic filter media have shown higher breathability than cellulose pulp filters (Chua *et al.* 2020). Fibrillation of nanocellulose fibers can be accomplished by wet beating, wet forming the nanocellulose filter, then freeze drying. The freeze drying process retains the fibrillation into the dry state and has shown matched breathability and filtration efficiency to N95 filters (Mao 2008). Zhang *et al.* (2019) demonstrated that the nanosized fiber diameter of an electrospun polyvinyl alcohol and cellulose nanocrystal composite filter significantly increased air permeability while increasing filtration efficiency.

Pore size and pore volume fraction

Filter permeability is highly governed by the pore size and pore volume fraction of the filter media. Achieving a filter composition with sufficient air permeability while not compromising filtration efficiency remains a challenge. For a filter dominated by deterministic size-exclusion filtration, high porosity and large pores increase permeability, but they decrease filtration efficiency. Due to nanocellulose's high capacity for functionalization and to form filters with high porosity and high specific surface area (Jiang and Hsieh 2015; Alavi 2019; Liu *et al.* 2021), nanocellulose technology helps optimize the balance between filtration efficiency and permeability. A layered filter composed of nanocellulose fibers coated by microcellulose fibers and protein nanoparticles demonstrated a filtration efficiency above 99.5% for PM1-2.5 as well as an extremely low pressure differential of 0.194 kPa/g (Fan *et al*. 2018). This layered filter achieved relatively high permeability due to the large pores produced by the microcellulose fibers and the high porosity granted by the nanocellulose fibers, while optimizing filtration efficiency by taking advantage of the high surface area of the nanocellulose layer as well as its ability for functionalization by the nanoprotein particles.

Mat thickness

While mat thickness increases filtration efficiency, it typically reduces permeability (Mao 2008). Mat thickness especially becomes an issue in particulate matter (PM) polluted air filtration, because to attain filter longevity and to combat PM plugging and cake filtration effects, a bulky, thick filter is needed, and increasing the thickness lowers air permeability (Chen *et al.* 2017). To combat these issues, a stainless steel screen (300 mesh, pore size of 48 micrometers) was coated with a thin cellulose nanofiber layer that was hydrophobically modified using polydimethylsiloxane. This filter exhibited high filtration efficiency for PM2.5 with high air permeability and high, long-term mechanical integrity under humidity range from 45% to 93% (Chen *et al.* 2017). Another way to address the issue of mat thickness's permeability reduction is *via* fibrillation of the nanocellulose fibers. Nanocellulose fibrils increase the surface area of the filter, lessening the thickness of mat required for equivalent filtration efficiency (Mao 2008). Nanocellulose's chemistry, ease for functionalization, and fibrillation capabilities render it a suitable filtration material to combat challenges such as mat thickness and its effect on air permeability and filtration efficiency.

Moisture-related effects

Studies suggest that as relative humidity increases, the flow resistance of fibrous filter media decreases with hygroscopic particle loading, but with non-hygroscopic particle loading, the higher relative humidity shows no effect (Gupta *et al.* 1993; Miguel 2003; Joubert *et al.* 2010, 2011). Montgomery *et al.* (2015) demonstrated this phenomenon with attachment of hygroscopic particles being irreversible in nature after exposure to high humidity. These findings indicated a physical transformation in the structure of the collected particles that does not reverse once the elevated humidity is lessened, and a permanent decrease in flow resistance with hygroscopic particle-loaded filter media. Other studies have shown that when wet particles collect on a filter surface, they conglomerate, continuously forming larger clumps of particles. This phenomenon decreases air permeability over time (Chen *et al.* 2017). Moisture can also cause wetted cellulose fibrils to clump and clog the pores of the fiber network, thus reducing air permeability (Mao 2008). These issues of moisture's effect on a filter's air permeability can be addressed by hydrophobic modification, which was covered earlier in this work under the section regarding moisture-related effects on filtration efficiency.

MANUFACTURING OPTIONS FOR CELLULOSIC MEDIA

Overview

This section will consider the use of layered structures, options for preparing the cellulose, various possible chemical treatments, ways to form plies of filter media, drying options, and possible post-drying hydrophobization of air filter media. Here the emphasis will be on operations and procedures. In many cases, these can be carried out with equipment that is commonly available in paper mills and related facilities.

Table 2 lists some texts and review articles covering aspects of papermaking technology that may be useful with respect to preparing filter media.

Table 2. Sources of Background Information Related to Preparation of Cellulose-based Filter Media

Topic area	Citation
Paper's resistance to wetting and the use of sizing agents	Hubbe 2007a
Factors affecting the uniformity of paper	Hubbe 2007b
Ways to impart hydrophobic character to cellulosic surfaces	Cunha & Gandini 2010a,b
Factors affecting the absorbency of paper products	Hubbe *et al.* 2013
The use of nanofibrillated cellulose in various applications	Khalil *et al.* 2014
Mechanical refining of cellulosic pulps	Gharehkhani *et al.* 2015
Wet-laid nonwovens manufacturing	Hubbe & Koukoulas 2016
Cellulose-based filter media	Junter & Lebrun 2017
Design aspects of fiber-based particulate filters	Jung & Kim 2020
Layered structure of N95 face masks	Wibisono *et al.* 2020
Manufacture of face masks using cellulose and nanocellulose	Garcia *et al.* 2021
Electrospun nanofibers for air filtration, *i.e.* bacterial cellulose	Lu *et al.* 2021
Ways to impart hydrophobic character to cellulosic surfaces	Szlek *et al.* 2022

Many specific examples of cellulose usage in filter media have been described in publications. In early work, Madsen and Madsen (1967) found that filters made from regenerated cellulose fibers (rayon) were able to match the filtration performance of polypropylene masks, for usage during surgery. Chien *et al.* (2018) compared Whatman filter papers with respect to the collection of sulfuric acid mists. The papers having smaller pore size were shown to be more effective, but at the expense of higher pressure drop. Dziubak and Dziubak (2020) observed 99.9% filtration efficiency of traditional air filters of the type used for automobile engine systems. Remarkably, there was a very great increase in separation efficiency with increasing mass loading of dust. In other words, the system appeared to be highly dependent on a cake filtration mechanism, in which collected dust itself was responsible for the subsequent filtration of particles from the air. Gustafsson *et al.* (2016, 2019) described a "mille-feuille" (a thousand leaves) paper product that had been prepared by a wet-laid nonwoven process. The media were found to be effective for effective removal of viruses from filtered water. Steffens and Coury (2007) studied the collection efficiency of cellulose-based HEPA filters for nano-sized aerosols. Yang *et al.* (2020) studied the use of *Juncus effuses* plant fiber media for particles in different size ranges.

A potential advantage of papermaking technology is the ease with which the composition can be adjusted as a means to achieve the properties required by specific kinds of products. For example, Keck and Wittmaack (2006) described a system in which a cellulose fiber was prepared by a process that they called denuding. This was used for precise sampling of semi-volatile inorganic particulates from the air. As illustrated in Fig. 21, the denuding process involved passage of the air successively through two "mini parallel-plate denuders" (MPPD), the first of which was coated with sodium carbonate (to collect acid gases) and the second of which was coated with phosphoric acid (to collect ammonia). Rojas *et al.* (1989) used secondary ion mass-spectrometry (SIMS) as a means to study the penetration of atmospheric aerosols through cellulose-based filters. The focus was on the filtration of dust associated with combustion. Zeng *et al.* (2019), as an alternative to using cellulose, prepared filter media from another wood component, lignin. The lignin-based aerogel filters were found to have a high efficiency of capture of fine airborne particulates.

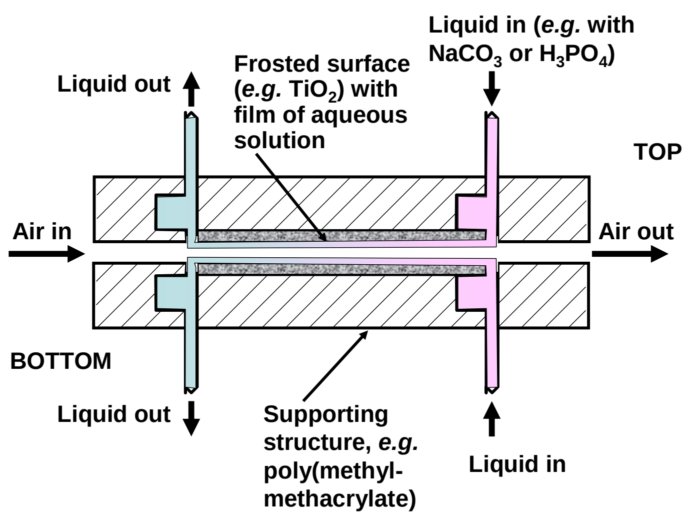

Fig. 21. The denuding process, which is designed to remove acid gases and ammonium from air as a means to be able to then collect semi-soluble particulates from air. Figure redrawn based on an original by Tsai *et al.* (2008)

Layers for Specific Purposes

Rather than having a thousand-layer structure, it will be proposed here that a better strategy may involve preparation of a limited number of layers, each of which has a defined role. As illustrated in Fig. 22, a certain layer may protect the remaining layers from moisture, it may absorb moisture, it may be designed to collect particles by an impaction mechanism, or it may be designed to filter out larger particles by a size-restriction-based sieving mechanism. The usage of multiple layers, each having a specific role, is shown for instance in the work of Wibisono *et al.* (2020). They studied a face mask in which a first layer prevented the penetration of fluids, the second layer retained viruses, and the inner layer absorbed fluids exhaled by the wearer of the mask. By contrast, Zangmeister *et al.* (2020) compared the filtration performance of cloth masks containing either synthetics, cotton, or blended fabrics. In that case, there appeared to be no difference in overall performance depending on whether a certain type of fiber was in a homogeneous layer or in a mixed layer.

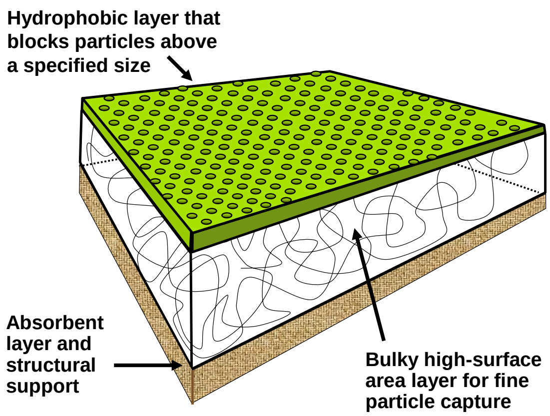

Fig. 22. Concept of a layered structure within a cellulose-based filter system in which one layer is specialized to resist liquid water, another is optimized to collect very small particles, and a third is designed to block large particles, absorb moisture, and provide strength to the structure

Moisture-protection barrier layer

Cellulose is naturally quite hydrophilic, as would be expected for a polymer having three –OH groups on each of its anhydroglucose repeating units. However, as noted in a recent review article (Szlek *et al.* 2022), those same –OH groups can provide a point of reaction and attachment for various hydrophobic functional groups. In addition, adsorbed hydrophobic materials, such as waxes, can be considered. As described in the cited article, systems involving ester bonds, reactions of tri-alkoxysilanes, and various plasma-related treatments are all promising ways to convert the surfaces of cellulosic materials to make them resist wetting by aqueous solutions. Though such modifications have been shown to be able to prevent surface wetting, they are not expected to block diffusion of water vapor or subsequent condensation of moisture within the material.

Moisture absorbency layer

Cellulosic fibers are known to be moderately effective absorbents for moisture. For instance, bleached softwood kraft fibers, *i.e.* fluff pulps, are known to imbibe about ten times their mass of water when used in absorbent products (Parham and Hergert 1980; Lund *et al.* 2012). Much higher ratios of water uptake can be achieved starting with cellulosic materials if they are treated in such a way as to prepare hydrogels (Hubbe *et al.* 2013).

There are basically two approaches to producing hydrogels from cellulosic materials. The conventional approach, usually starting with a relatively pure dissolving pulp grade of cellulose fibers, involves carboxymethylation. This process involves an etherization reaction that takes place at a high concentration of alkali with chloroacetic acid (Shui *et al.* 2017). The resulting carboxymethylcellulose (CMC) can be then cross-linked to prepare a hydrogel. Such materials can earn the label of "superabsorbent" in cases where they are able to take up more than about ten times their dry weight in aqueous solution (Kabriri *et al.* 2011), though ideal formulations can reach values of greater than 1000 parts of imbibed aqueous solution in comparison to dry weight (Hubbe *et al.* 2013). CMC has been well demonstrated as a component of superabsorbent polymer (SAP) hydrogel formulations (Oppermann 1995; Bao *et al*. 2012). However, due to their more favorable cost-to-performance ratio, petroleum-based SAPs are typically used in such products as disposable diapers and incontinence pads.

A second way that cellulosic material can be converted into a hydrogel is by intense and protracted mechanical action in the wet state. The product of such action has variously been called microfibrillated cellulose (Turbak *et al.* 1983; Lavoine *et al.* 2012), nanofibrillated cellulose (NFC) (Khalil *et al.* 2014; Lindström 2017; Naderi 2017; Zambrano *et al.* 2020), and cellulose nanofibril (Benitez and Walther 2017), among other terms. Though a NFC suspension of sufficient concentration (*e.g.* 2% solids or more) may behave like a gel, typical applications of gels require that the material stay together as a unit even when diluted. This can be achieved by means of crosslinking (Spoljaric *et al.* 2014; Purkayastha *et al.* 2022). By adjusting the level of crosslinking, the formulator can trade away some of the absorption capacity in favor of the strength of the hydrogel structure, depending on the requirements of the application.

Though the citations given above provide guidance to the preparation of hydrogels, typical air filtration applications would require the availability of the hydrogels in a dry form. Indeed, a layer of swollen hydrogel would be expected to be very effective in blocking the flow of air, rendering the filter medium impermeable. Thus, the amount of moisture that can be absorbed in an effective air filtration system, employing SAPs, may be limited by the tendency for blockage if the sum of the amount of SAP times its degree of swelling is too high. In applications where the goal is to absorb liquids, such problems can be overcome by use of relatively large cellulosic fibers or other structures to allow channeling of fluids within the material (Hubbe *et al.* 2013). However, some such strategies would be incompatible with the high standards of fine particle collection efficiency that are needed in some air filtration equipment.

Impaction barrier layer

As described earlier in this article, the smallest particles to be collected during the filtering of air are most likely to be retained by their diffusion onto the solids surfaces as they pass through the media. Thus, to achieve a high efficiency, a high amount of surface area needs to be provided within the filter device. According to the Kozeny-Carman equation (Kozeny 1927; Carman 1937; Carrier 2002), when all other terms of held constant, a high surface area will imply a relatively high resistance to flow. This is evident from the following form of that equation,

$$k = \frac{\varepsilon^3}{k_c S^2 (1-\varepsilon)^2} \tag{15}$$

where ε is the fractional void volume (or porosity) of the packed bed, k_c is essentially a correction factor, which includes *e.g.* the effect of tortuosity, and S is the specific surface area of the solids. In principle, the adverse effect on permeability can be mitigated by aiming for structures having a low apparent density of a layer that contains nano-scale fibers, taking advantage of the high exponents associated with the fractional void volume terms present in the Kozeny-Carman equation. Strategies for creating a very high surface area of cellulosic material (*i.e.* nanocellulose) and avoiding its compaction in the course of drying will be considered in later subsections. An additional challenge that will be discussed later is how to avoid the densification of such a layer that might result from its moistening, resulting in a loss of stiffness of the material.

Support layer and size-based sieving

The impaction barrier layer, as just described, can be expected to have low strength, making it vulnerable to abrasion. Fortunately, when one considers the attributes that would be needed in a size-restriction barrier layer, such a layer has potential to serve as an outer, protective layer. As shown earlier in this article, there is a transition point at a particle diameter of about 100 nm, above which the diffusion mechanism of particle capture can be expected to become less effective (Lee and Liu 1980). Especially when the flow velocity is high (Liu and Lee 1976), it is important to be able to capture those larger particles by means of a filter layer that functions in the manner of a screen. Later sections will consider options such as fiber selection and choice of refining levels in order to achieve such goals in a papermaking operation.

Options for Preparing the Cellulose

Several categories of different preparation methods for cellulosic materials can be considered, depending on the details of the type of filter media to be produced. These range from conventional mechanical refining of pulp fibers, much more extensive mechanical action (often supplemented by chemical pretreatments) to make highly fibrillated products, including nanofibrillated cellulose, and a variety of treatments that change the chemical composition. Some of these options have been discussed by Garcia *et al.* (2021) in their review article on the manufacture of face masks. Also, there are some options involving blending of cellulosic materials with other fibers or with minerals.

Selection of cellulosic fiber type

Depending on the desired coarseness, cellulose-based filter media can be prepared from eucalyptus kraft, other hardwood kraft, softwood kraft, cotton linter, various bast fiber types, or regenerated cellulosic fibers such as rayon (viscose). Some of these options are depicted in Fig. 23, which contrasts the typical sizes and shapes. The fiber types have diameters generally in the range from 15 to 100 μm.

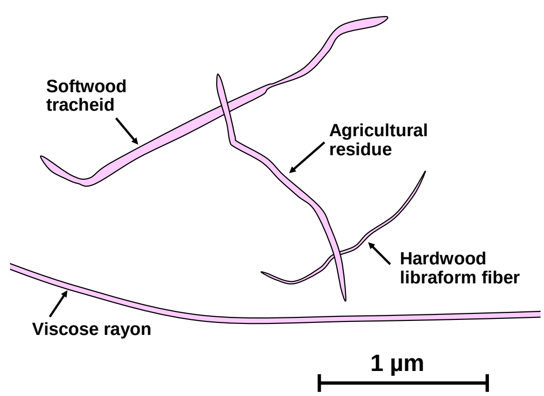

Fig. 23. Morphology of typical cellulosic fibers that can be considered for the preparation of filter media. The softwood, hardwood, and agricultural fiber examples are drawn from micrographs provided by Sood and Sharma (2021). Rayon fiber diameter is drawn based on the intermediate value tested by Graupner *et al.* (2018).

In cases where such fibers are subjected only to low levels of mechanical refining, the distribution of pores between the fibers within filter paper will be a strong function of the diameters of those constituent fibers.

Cotton is readily available in two size ranges. Figure 24 illustrates a bowl of cotton, in which both types of fibrous material are shown. Textiles, including some wet-laid nonwoven products, are mainly produced from staple cotton fibers (Sczostak 2008). These are about 20 to 45 mm long, thin-walled, and about 12 to 22 μm in diameter. The length is much too great to be handled in a conventional paper machine system. A smaller category of cotton material, called linters (Sczostak 2008), become separated during processing of cotton. These have a length of about 2 to 6 mm, which permits processing with ordinary papermaking equipment, especially after moderate cutting of the length of the longest fibers and by bypassing any screens in the approach flow to a paper machine. Cotton linters are about 17 to 27 μm in diameter. The fiber cell walls are relatively thick, which can contribute to the stiffness of the fibers, leading to a bulky, porous paper structure. Ward *et al.* (1965) reported the effects of different levels of mechanical refining in the properties of cotton linter pulp for papermaking.

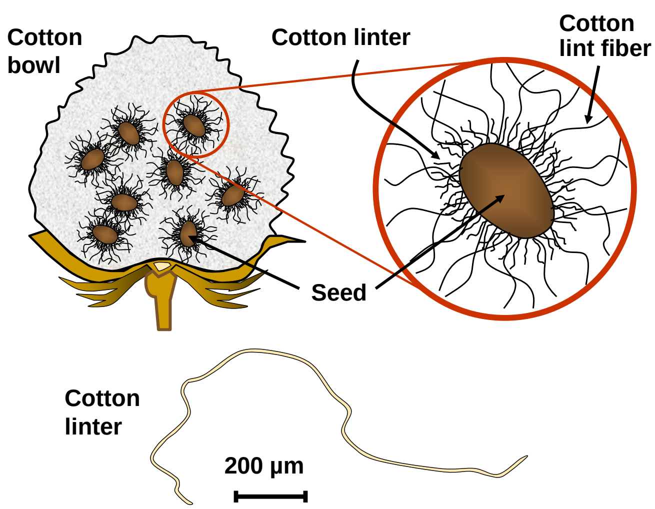

Fig. 24. Illustration of cotton lint (fibers) and linters present in a bowl of cotton. The illustration of the cotton bowl and its contents are redrawn based on an original from the Dieu Donné hand papermaking company (https://aboutabeautifulbook.wordpress.com/2015/02/28/papermaking/). The dimensions of the individual cotton linter are based on an SEM micrograph from Luo *et al.* (2013).

A study by Hosseini and Tafreshi (2011) showed that the performance of air filter media can be affected by the cross-sectional shapes of the component fibers. Angular cross-sections, such as square, were found to contribute to higher resistance to flow in comparison to rounded cross-sections, which are called "streamlined" in the article. The effects were deemed important in a so-called slip flow regime, wherein the fiber diameter is of a similar order of magnitude to the mean free path of gas molecules (*e.g.* 65 nm).

Payen *et al.* (2012) showed that the use of blends of different kinds of fibers can offer advantages relative to the performance of air filters. Decreasing fiber diameter generally resulted in increasing collection efficiency but decreasing air permeability. It was found that combining strongly contrasting fibers yielded favorable collection efficiency at given levels of resistance to permeation. Onur *et al.* (2018) showed that the permeability and pore size distribution of a highly fibrillated cellulose structure could be adjusted by selected addition of perlite, along with the use of a wet-strength agent during sheet formation.

Conventional refining

When papermaking fibers are subjected to repeated compression and shearing in a refining operation, they become progressively more conformable (Gharehkhani *et al.* 2015). The key actions within a conventional fiber of the type used by papermakers are illustrated in Fig. 25. As shown, the mechanical action causes the wet fibers to swell due to internal delamination, and the outer layers become fibrillated and partly detached as cellulosic fines. The relative bonded area within the resulting paper is increased by refining, leading to a denser structure. In principle, by selecting the extent of refining, the papermaker has means to adjust the tightness of the pore structure.

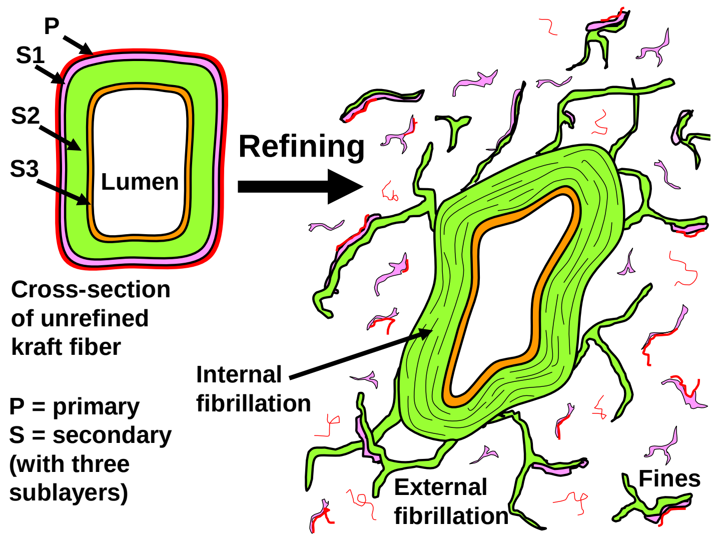

Fig. 25. Action of a mechanical refiner that can be used in a papermaking operation to fibrillate kraft or cotton cellulose fibers, or if used with many multiple passes can be used to prepared microfibrillated cellulose (less fibrillated than NFC). Figure adapted from Debnath *et al.* (2022)

In a typical refiner system, of the type used in the development of kraft fibers for printing grades of paper, the unrefined fibers enter at a solids content (consistency) of about 4% to 5%. The consistency is high enough that each fiber will spend most of its time somewhat entangled with neighboring fibers in the suspension. The flocky suspension passes in the outward direction between a rotating disk and a stationary disk, where each of the disk surfaces has a pattern of rectangular bars. By forcing the fibers momentarily together and shearing them multiple times, the refining action causes delamination within the cell walls. The outer fibers layers (P, S1, and usually part of the S2 sublayer) will become pealed outward from the fiber surfaces, creating detached cellulosic fines and still-attached fibrillation of the fiber surfaces.

Mao *et al.* (2008) described the use of a high, but conventional level of refining to create filter media for N95 masks that performed similarly to their commercially available counterparts. The refined fibers were partially freeze-dried as a means of adjusting the degree to which fibrils on the fiber's surfaces remained extending outwards from the fibers. It is well known that conventional drying will cause external fibrils to lie down tightly against the surfaces to which they are attached. Such a matting down of fibrils during drying (and other morphological changes) can cause the specific surface area of the highly refined fiber to revert almost to its original value before refining was started (Kang *et al.* 2018).

Micro- and nanofibrillated cellulose

The need to remove very small particles, *e.g.* diameter less than 300 nm, from air provides a motivating factor to consider the use of nanocellulose. As described in review articles, intense and prolonged mechanical action provides the main path towards the production of nanofibrillated cellulose (NFC) and related products (Khalil *et al.* 2014; Lindström 2017; Naderi 2017; Zambrano *et al.* 2020). When mechanical processing is the only tool employed to produce nanocellulose, the energy consumption may fall in the range of 20,000 to 30,000 kWh/ton (Siró and Plackett 2010; Khalil *et al.* 2014). Substantial savings in energy, as well as in the time of mechanical processing can be achieved by pretreatment. For instance, Siró and Plackett (2010) stated that certain pretreatments may decrease the required energy to about 1000 kWh/ton. Effective treatments to reduce energy requirement include cellulase enzymes (Pääkkö *et al.* 2007), phosphorylation (Lindström 2017), periodate oxidation (Tejado *et al.* 2012), and TEMPO-mediated oxidation (Chaker *et al.* 2014; Nemoto *et al.* 2015; Rol *et al.* 2017) of the cellulosic surfaces. The article by Rol *et al.* (2017) showed that even more energy can be saved by using twin-screw extrusion to bring about fibrillation at a high solids content. Figure 26 depicts three main types of cellulose products that can be considered, depending on the requirements.

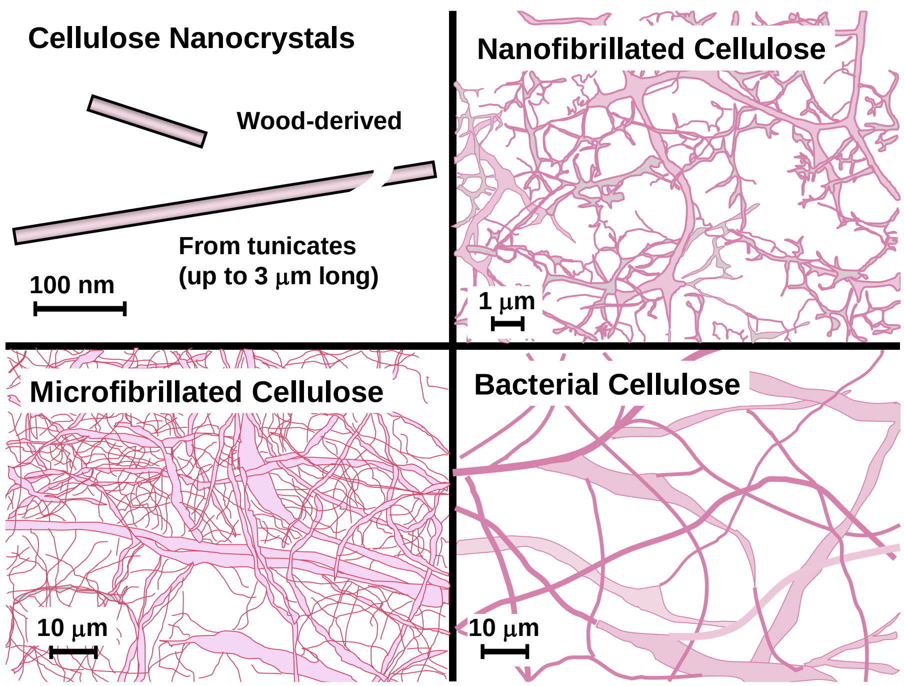

Fig. 26. Steps in the preparation of nanocellulose, including pretreatment, application of strong and protracted hydrodynamic shear (to make nanofibrillated cellulose) or concentrated acid solution treatment (to make cellulose nanocrystals). The images for cellulose nanocrystals, nanofibrillated cellulose, and bacterial cellulose were first published by Hubbe *et al.* (2017). The image representing microcrystalline cellulose was drawn based on a micrograph from the Borregaard company.

The structure of NFC can be markedly different, depending on the method of preparation. A typical NFC product made by mechanical processing alone can be expected to have a highly branched structure with a wide range of widths within the fibrillar structure (Hubbe *et al.* 2017). By contrast, products having narrower and longer fibrils often result when the starting material has been pretreated so as to favor easier fibrillation. For instance, the work of Pääkkö *et al.* (2007) demonstrated a further potential advantage of employing enzyme pretreatment, in addition to the energy saving. They were able to obtain almost uniformly narrow, unbranched fibrils having narrow diameters of about 5 to 6 nm. Though the cited authors were particularly interested in the building of strong gels, one could envision that a combination of narrow fibril diameter and unbranched nature might contribute to high performance of an impaction-type filtration layer, as was discussed. The choice of shearing device used to defibrillate the material also can be expected to influence the morphology. In particular, a structurally diverse material can be expected when using a micro-grinder (Nair *et al.* 2014; Velazquez-Cock *et al.* 2016), in comparison to when using a high-pressure homogenizing system (Lee *et al.* 2009; Besbes *et al.* 2011; Dhali *et al.* 2021). In principle, with the selection of pretreatment conditions and the means of applying hydrodynamic shear, an engineer developing nanocellulose filter media will have many options to control the resulting structures.

Various researchers have reported on the performance of air filter media prepared with NFC and related materials (Nemoto *et al.* 2015; Omori *et al.* 2019; Zhang *et al.* 2020). Fan *et al.* (2018, 2019) achieved a high collection efficiency at relatively low pressure drop in systems that contained NFC and protein nanoparticles. Zhang *et al.* (2020) reported that the addition of about 0.8% of NFC increased the collection efficiency of particulates without increasing resistance to flow. Omori *et al.* (2019) surprisingly reported that collection efficiency decreased with increasing nanofiber addition to a microfiber structure. Skaria *et al.* (2014) found that increasing the amount of nanofibers in a face mask resulted in increased pressure drop. This contributed to greater leakage past the face seal, thus defeating the purpose of increasing the collection efficiency. The review article by Liu *et al.* (2017) stated that nanofiber structures are able to outperform other filter media for the interception of fine particulates at a stated level of pressure loss.

Bacterial cellulose

The use of bacterial cellulose (BC) as a source of NFC offers some potential advantages. Most importantly, BC is listed as generally regarded as safe (GRAS) by the US Food and Drug Administration. This may be important in applications where ingestion of some of the material seems likely. As shown in Fig. 27, cellulose is continuously biosynthesized at the surface of cells such that groups of six macromolecules are able to form elementary microfibrils. Because BC is synthesized essentially in nano form in bacterial cultures, much less mechanical energy will be needed in typical cases to prepare the NFC. The downside is that bacterial cellulose is much less available and more costly compared to cellulose pulp obtained from trees (Esa *et al.* 2014; Azeredo *et al.* 2019).

Results of air filtration using media prepared with BC have been reported. Gustafsson and Mihranyan (2016) used BC to tailor the pore size distribution in filters. By adjusting the temperature and rate of drying in a hot-press, they were able to tune the characteristic pores sizes in the range of 10 to 25 nm. Thereby, they were able to achieve a high efficiency of virus retention. Liu *et al.* (2017) reported a high collection efficiency and a high air penetration rate when using media comprising BC and soy protein isolate.

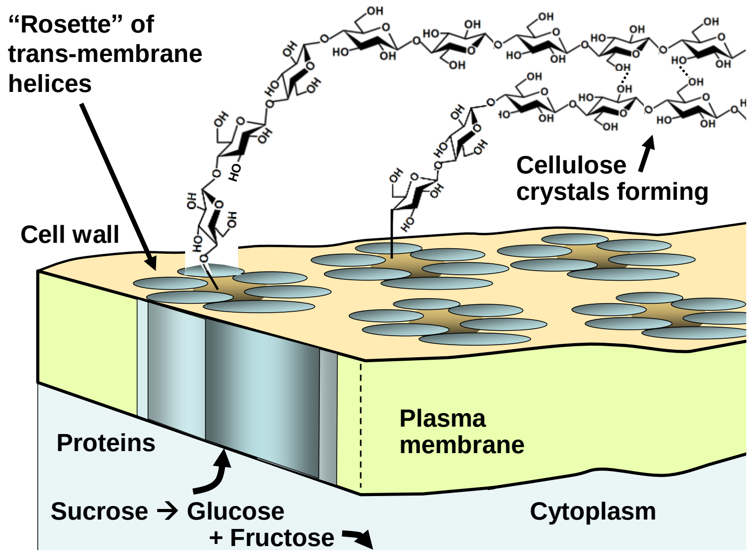

Fig. 27. The biosynthesis process of cellulose, leading to (for instance) the development of bacterial cellulose

Regenerated cellulose

To further extend the range of possible fibril diameters, lengths, and other features, it is well known that cellulose solutions can be regenerated into fiber form by a process of drawing and passage through a coagulation bath. Filaments prepared by regeneration of cellulose have been shown to be effective for air filtration, usually in the form of wet-laid nonwoven mats (Madsen and Madsen 1967). In light of these cited promising results with regenerated cellulose filter media (rayon), there appears to be a need for current research in this area. The basic process is depicted in Fig. 28.

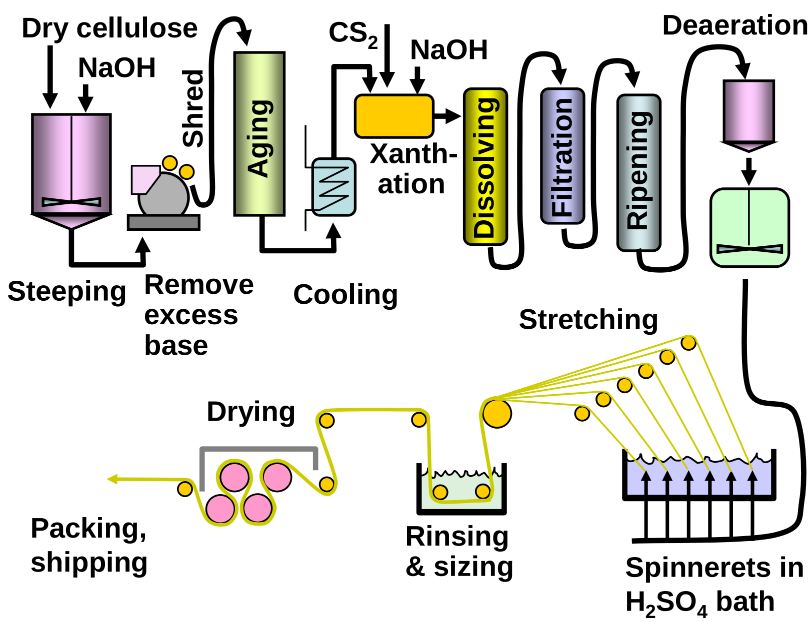

Fig. 28. The regeneration of cellulose to obtain fibers (viscose rayon process)

Mercerization, which is a common treatment applied to cellulose in the filter paper industry (Liu *et al.* 2015), involves immersion of the material into moderately concentrated NaOH (*e.g.* 22%) (Stana-Kleinschek *et al.* 2004). Such treatment causes the cellulose chains to rearrange themselves, changing from a native cellulose I crystalline form into a cellulose II form, which is in common with fully regenerated cellulose products. Though the general dimensions and shape of the native cellulose fibers are preserved, the fiber surfaces become smoother (Stana-Kleinschek *et al.* 2004; Obendorf 2004).

Woo *et al.* (2011) considered another chemical modification of cellulose, conversion to the dialdehyde form. They evaluated the resulting product as a filter medium for airborne and waterborne bacteria and viruses. The treatment was found to decrease the pressure drop during filtration, compared to untreated fibers, and it was more effective for removal of the microbes. Dialdehyde cellulose has a tendency to become highly swollen in water (Dalei *et al.* 2022). A tendency of dialdehyde groups to form hemiacetal linkages appears to contribute to the insolubility of the material, despite its swelling tendency. The hydrogel character of dialdehyde cellulose also may make it a suitable candidate for use in a moisture absorbent layer of a filter, as discussed earlier.

Electrospinning

Though the technology is based on cellulose regeneration, electrospinning merits separate discussion due to some unique attributes (Lu *et al.* 2021). As illustrated in Fig. 29, the electrospinning of cellulose and various derivatives of cellulose resembles ordinary regeneration insofar as one starts with a solution, a filament can be continuously drawn, and one ends up with a solid filament (or some other shape, depending on the application). Electrospinning differs in that a strong voltage difference is imposed between the nozzle zone and a target area. Electrostatic forces bring about a rapid stretching of the filament, such that very small diameter material can be formed. Rather than attempt to collect the material on spools, usually the produced nanofilament material is allowed to build up as a jumbled layer on the charged target surface.

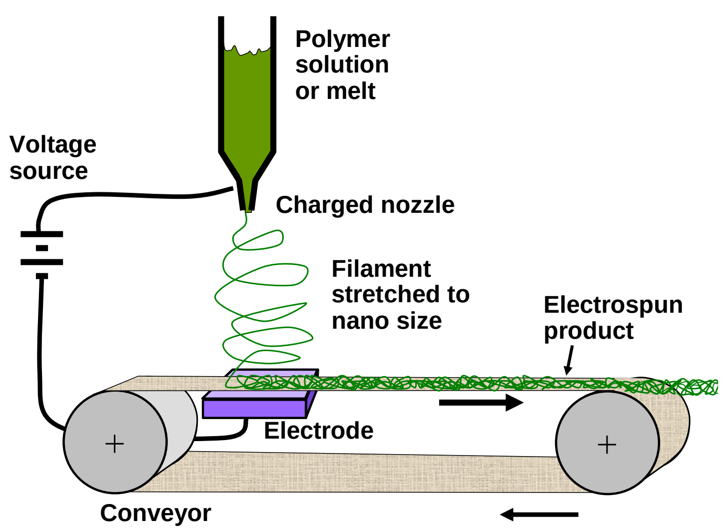

Fig. 29. Schematic of an electrospinning process to prepare very narrow filaments of cellulose or other polymer capable of being placed into solution and then greatly stretched during drawing

Kadam *et al.* (2018) and Li *et al.* (2019) reviewed the topic of electrospun nanomaterials in their use in air filtration. High filtration effectiveness of such materials was attributed to the narrow diameters of the fibers, the small pore sizes, and the high surface areas. The article also considered various surface treatments and their effects on filtration performance. Most of the examples mentioned in the cited reviews were based on polymers other than cellulose. For example, Hung and Leung (2011) showed a case where nanofibers having a diameter of 94 nm achieved almost four times higher filtration efficiency (> 38% for the most problematic particle size of about 100 nm) compared to a filter with 185 nm diameter nanofibers (> 11% for 120 nm particles). Fan *et al.* (2018, 2019), whose work already was mentioned in the context of bacterial cellulose, used electrospinning of zein protein as a means to prepare the supporting structure of their filter system. It was found, however, that the electrospun versions of filter media resulted in a higher pressure drop in comparison to an emulsion-based mode of filter preparation (Fan *et al.* 2019).

Cellulose-based and related electrospun systems and their usage in filter media were reviewed by Lv *et al.* (2018). Only a few systems involving the electrospinning of cellulose were cited in that work (Ma *et al.* 2005; Kim *et al.* 2006b; Awal and Sain 2012; Chattopadhyay *et al.* 2016). Kim *et al.* (2006b) showed that cellulose that had been dissolved in either an ionic liquid (LiCl/NN-dimethyl acetamide) or N-methylmorpholine oxide (NMMO) could be electrospun successfully when using an aqueous bath for regeneration. The results could be adjusted by controlling the temperature, flow rate, and the distance between the nozzle and collector. Fibers having diameters in a range of 250 to 750 nm were obtained. The material prepared from NMMO had a typical level of cellulose crystallinity (42% to 66%), whereas the material prepared for ionic liquid solution was non-crystalline. Unpublished work (Hubbe 1979) showed that an NMMO solution of cellulose, when formed into droplets, had an undesired tendency to spread out and form a congealed film on the surface of a water bath. A figure included in the work of Kim *et al.* (2006b) appears to show related behavior, since the product resembled a porous continuous membrane rather than a mat of nanofibers. Notably, the electrospun material reported by Ma *et al.* (2005), Awal and Sain (2012), and Chattopadhyay *et al.* (2016) had been acetylated, meaning that the product was electrospun cellulose acetate rather than cellulose itself. Many studies have incorporated cellulose nanocrystals CNC into various electrospun polymer systems (Vallejos *et al.* 2012), and most notably this has been achieved with CNC particles incorporated into a regenerated cellulose matrix (He *et al.* 2014). Based on the large amount of work being carried out with electrospinning, but only a relatively small number of publications incorporating cellulose and its derivatives, it appears that there is a need for further research in this area in the future.

Fiber blends: Synthetics with cellulosics

Blends of different kinds of fibers often can help to meet a diverse range of product requirements, such as strength and capture efficiency. Table 3 lists some of the blends that have been considered. In addition, Payen *et al.* (2012) have reviewed earlier work related to the use of fiber blends for filter media. In one of these examples (Hui *et al.* 2018), it was shown that bleached kraft pulp fibers have the potential to provide absorbency, softness, and lower cost, whereas longer synthetic fibers such as polyethylene terephthalate (PET) can provide resistance to tearing.

Table 3. Studies Involving Blends of Cellulose and Other Fiber Types for Air Filtration Media

Cellulose Type	Second Type of Fiber	Citation
Various common cloth	Polyester, *etc*.	Rengasamy *et al.* 2010
Softwood kraft pulp fibers	Polypropylene (PP) and polyester (PET)	Hui *et al.* 2018
Nanofibrillated lyocell	Coated on filter paper (also cellulose)	Long *et al.* 2018
Kapok (waxy) fibers	Hardwood cellulosic fibers	Sun *et al.* 2018
Cellulose nanocrystals	Electrospun poly(vinyl alcohol) (PVOH)	Zhang *et al.* 2019
Cotton cloth blends	Polypropylene	Zangmeister *et al.* 2020

Zeolites

Another kind of blending worth considering is the incorporation of mineral or other particles in a cellulose-based filter media. Zeolites can be a promising component of air filters due to their exceptional capacity to take up monomeric contaminants, such as oils and volatile organics (Su *et al.* 2018). Ma *et al.* (2018a) also demonstrated antimicrobial performance when zeolites were combined in an air filtration system with softwood kraft cellulose fibers.

Options for Chemical Treatments

For purposes of air filtration, especially in the case of face masks, three important categories of chemical treatment can be applied, namely wet strength resins, hydrophobic sizing agents, and antibacterial treatments. In addition, some such treatments also could be expected to affect triboelectric properties.

Wet strength agents

As has been mentioned, a likely role of cellulosic material in an air filtration system can be to absorb moisture. Especially when a filter will be exposed to human breath, the material must be able to withstand moistening without coming apart. In cases where such moisture absorption is regarded as desirable, papermakers often will employ a wet-strength treatment. The most widely used wet-strength agent, which cures best under alkaline pH conditions of manufacture, is the poly(amidoamine epichlorohydrin) (PAAE) type of resin (Espy 1994; Lu *et al.* 2020). Positively charged functional groups, which include amine groups and azetidinium groups, favor efficient adsorption and retention of the PAAE on fiber surfaces at the point of mixing with the fiber suspension. During drying of a paper product, the PAAE can react in two ways, both involving the azetidinium groups. In cases where there is a significant level of carboxylic acid groups on the fiber surfaces (which will be related to the hemicellulose component), the PAAE can form covalent bonds with those groups. In addition, the PAAE can react with itself, leading to a hardening of the resin (Espy 1994, 1995). These steps are shown in Fig. 30. The resulting covalent bonds, in each case, are tolerant of the presence of water, which often allows the product to retain substantial strength even after complete soaking. Although PAAE is widely regarded as a wet-strength agent, the treatment also will increase the dry strength of the paper.

Ji *et al.* (2019) used a mixed-component wet-strength formulation when preparing filter paper for the air intake of automobile engines. This was described as a water-based epoxy resin emulsion, but it also contained o-cresol and formaldehyde, which is a well-known acid-curing wet-strength system. The treatment added to the wet strength of the filter material, and it also contributed some water-repellency, while maintaining the filtration performance.

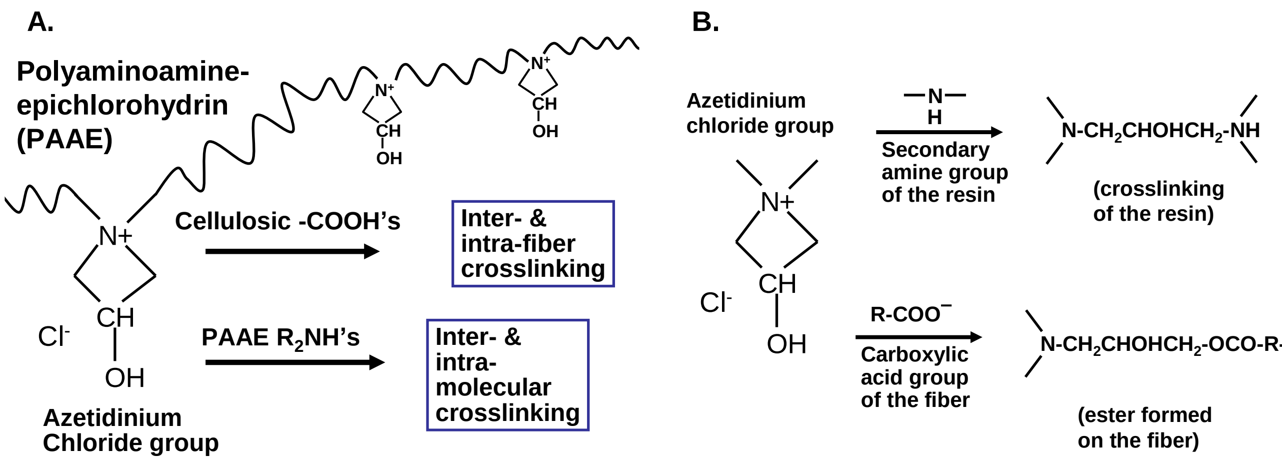

Fig. 30. Chemistry of the polyamidoamine-epichlorohydrin wet-strength resin system

Gustafsson *et al.* (2019) reported the use of a different kind of reagent to achieve wet strength in a filter paper product designed to retain viruses. The reagent citric acid was catalyzed by sodium hypophosphite, with curing for 12 h at 80 °C. In principle, a molecule such as citric acid having multiple carboxylic acid groups can form multiple ester bonds with –OH groups at the surfaces of cellulosic fibers. When two such bonds are formed by one molecule of citric acid, a crosslink point has been created. By contrast, unreacted carboxylic acid groups, due to their polar nature, will contribute to water affinity. Accordingly, different degrees of hydrophilicity *vs.* crosslinking can be achieved by adjusting the temperature and time of heat-curing. Notably, the filter product being considered by Gustafsson *et al.* (2019) was intended to function under wet conditions, but the results are relevant to air filter media that may become wet.

Hydrophobic sizing agents

Papermakers use the term internal sizing agent to denote treatments that cause paper products to develop hydrophobic character as the paper is being dried. Different classes of sizing agent are generally employed, depending on whether the paper is being produced under acidic ($4 < pH < 5.5$) or alkaline ($7.5 < pH < 9$) pH conditions (Hubbe 2007a; Ehrhardt and Leckey 2020). Under acidic papermaking conditions, rosin products are employed, always with the sequential addition of aluminum sulfate (papermaker's alum) or a related product. Under alkaline conditions, either alkenylsuccinic anhydride (ASA) or alkylketene dimer (AKD) is employed. Figure 31 shows the main features of treatment with ASA. These agents are added as an emulsion that usually is stabilized by a cationic polymer, and the charge provides efficient retention onto fiber surfaces. Further information about ways to render cellulosic fibers hydrophobic has been described in review articles (Khalil *et al.* 2014; Szlek *et al.* 2022).

Heydarifard *et al.* (2016) prepared what they called a hydrophobic filter product by use of an aqueous mixture of glutaraldehyde and zinc nitrate, together with poly(vinyl alcohol). The treated filter paper was cured for 30 minutes at 120 °C. The dry and wet strength properties of the paper were improved. Chen *et al.* (2017) incorporated the hydrophobic agent poly-dimethylsiloxane (PDMS) to prepare hydrophobic NFC. The PDMS was transferred as vapor to the cellulosic surface by heating the PDMS to 50 °C for 4 h.

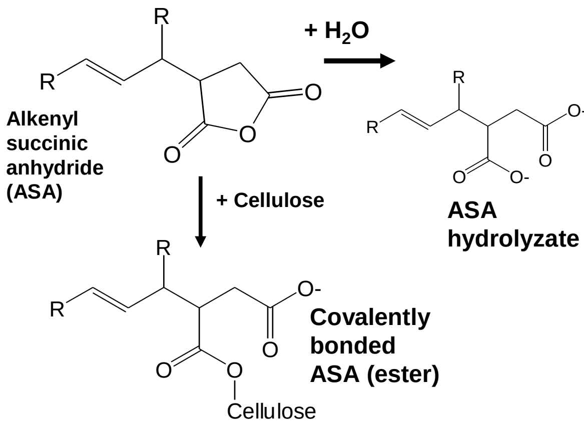

Fig. 31. Schematic of treatment with alkenylsuccinic anhydride (ASA) to render cellulosic surfaces hydrophobic

Antibacterial treatments

Various antibacterial treatments have been considered to inhibit the growth of microbes, especially in the case of facemasks. There is a concern that facemasks, due to the moistness of breath, could maintain the collected microbes in viable conditions, perhaps leading to their transmission to others (Delanghe *et al.* 2021). Also, it is clear that facemasks are likely to collect exhaled bacteria and viruses (Hu 2022). The breath was shown to contain aerosol droplets, and these contribute not only to transport of microbes but also to keep the mask material moist.

A review by Garcia *et al.* (2021) provides an extensive overview of the use of antibacterial materials in cellulose-containing face masks. These were listed as natural bioactive compounds (for instance terpenoids), metals (such as silver nanoparticles), and various amines, including quaternary ammonium compounds, among others. Expected effects of such substances are depicted schematically in Fig. 32. As in the case of chitosan, which can be obtained by alkali treatment of crustacean shells, many of the effective agents have a cationic charge.

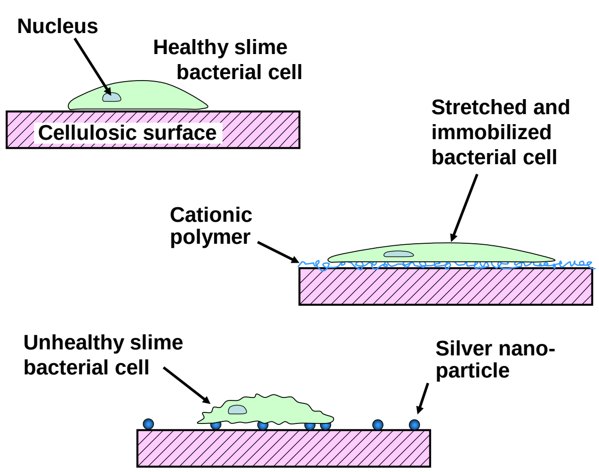

Fig. 32. Examples of antibacterial substances that have been shown to be effective when incorporated into cellulose-based filter media

Various publications have reported antimicrobial activity in filter media that had been treated with suitable agents. For example Tiliket *et al.* (2011) treated facemask material with poly(ethyleneimine) (PEI), which is highly cationic at intermediate to low pH values. The PEI treatment was shown to increase the retention of T4D bacteriophage. Imani *et al.* (2011) employed both chitosan and nanosilver particles with polyacrylic acid on filter paper as a means of achieving antibacterial effects. Ma *et al.* (2018a) demonstrated antibacterial effects in air filters that had been treated with metal-organic frameworks, *i.e.* zeolitic imidazolate.

Options for Forming Filter Media Layers

Once decisions have been made about the materials and chemical treatments to be employed in preparing filter material, there are still some options to consider regarding how the components will be put together in a manufacturing process. Though the emphasis here will be on paper technology (either single ply or multiple plies), one should not rule out air-laid forming, which is usually classed as a textile technology.

Paper forming

The paper forming process can be envisioned by imagining a flow of suspended fibers arriving one by one at the surface of a mat that is ultimately supported by a screen (the forming fabric). Figure 33 shows the basic steps, using typical equipment designed for wet-laid nonwoven forming (Brandon *et al.* 1980). Due to the high ratio of length to thickness of typical papermaking fibers (aspect ratio of 50 to 100), each arriving fiber will tend to lie down approximately in a planar manner relative to the plane of the forming fabric. A more realistic model of the process would take into account the fact that substantial flocculation of papermaking fibers is unavoidable due to crowding within typical solids content (consistencies) during formation on typical paper machines (Kerekes and Schell 1992). Nevertheless, it remains true that conventional paper sheets can be approximated as a bunch of co-planar layers. The fact that such a structure can be suitable for filtration media is obvious from the widespread usage of filter papers, which are mostly produced using conventional paper forming processes.

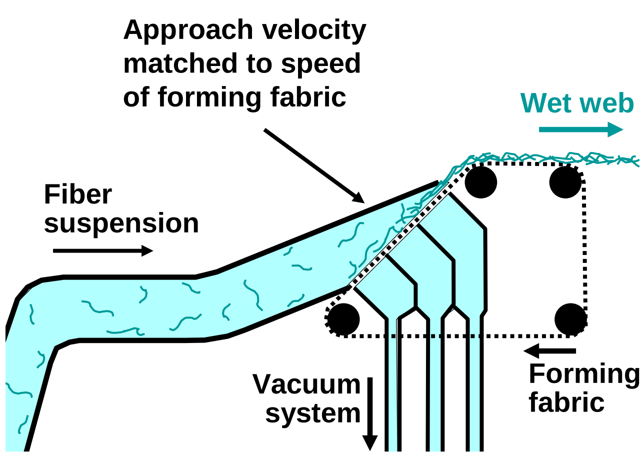

Fig. 33. The paper forming process, in which a suspension is dewatered on a continuous screen (in this case an inclined Fourdrinier forming device), pressed, and then dried by evaporation. Figure redrawn from US Patent 4,200,488 by Grandon *et al.* (1980)

When fibers longer than about 4 mm are used in a papermaking process, the term "wet-laid nonwoven" is often used. Some specialized methods need to be used due to the much greater tendency of such long fibers to form clusters and knots. Whereas a headbox consistency of about 0.5% would be regarded as typical for conventional papermaking, wet-laid nonwovens processes often involve consistencies up to ten times lower. In addition, mucilage (often a very high mass anionic copolymer of acrylamide) is used in sufficient quantity to raise the viscosity of the aqueous phase (Hubbe and Koukoulas 2016). A favorable feature of the wet-laid forming process is that the component fibers tend to move toward more open areas of the initially formed mat on the forming fabric; this "healing effect" contributes to a more uniform pore size that one might expect based on random deposition of the fibers (Wrist 1962; Gorres *et al.* 1986; Norman *et al.* 1995; Hubbe 2007b; Hubbe and Koukoulas 2016). This effect also contributes to a more uniform distribution of pore sizes within a mat. At the same time, some non-uniformity is present due to unavoidable entanglements and flocculation among fibers.

Chemical treatments in the course of papermaking, as well has processes such as refining of the fibers and wet-pressing of the sheet before it is dried, offer opportunities for the papermaker to make adjustments in the bulk (reciprocal of apparent density) and air permeability of paper. For example, it is known that sequential treatment of papermaking stock with a cationic polymer (cationic starch or cationic retention aid) and a nanoparticle (colloidal silica) can favor more rapid dewatering during paper formation (Andersson and Lindgren 1996; Hubbe 2005). This relationship implies that the wet web of paper, due to the treatment with the cationic polymer and colloidal silica, has a more bulky and porous structure. In ordinary papermaking, much of that increased bulk is seldom noticed by the customer, since strong compressive forces are exerted during wet-pressing of the paper before the dryer section of the paper machine. Many grades of paper are also calendered, which involves passing them between hot, smooth rolls at high pressure. When preparing filter paper, the production team has options to reduce or eliminate such densifying steps in the process, thereby having a means to control the pore size distribution within the product.

Multi-ply forming

Earlier in this article it was noted that there is often an advantage of providing different layers of filter media, each having a different specialization. For instance, a layer with very high specific surface area, tiny fibril size, and large proportional void space could be prepared as a means of maximizing diffusional interception of very small (say less than 300 nm) particles. But such a layer would be weak and would benefit from support as well as outward protection by sturdier layers. In addition, as noted earlier, another layer could specialize on the physical blocking of particles larger than about 300 nm. In principle, such multilayer structures can be achieved by mature papermaking technologies including ordinary papermaking and coating. Multi-ply formation of paper is a mature technology (Nordstrom 2016). Figure 34 suggests how such a process might be implemented by means of two or more plies formed in separate papermaking processes.

Zhang *et al.* (2002) describe a study in which an NFC layer was essentially coated onto a base ply of filter medium. This type of application takes advantage of having the absorptive capability of the base ply to draw water out of the NFC suspension during the coating process. Such dewatering of the coated material, as it is applied to a based ply, will tend to immobilize the material, due to an increasing solids content (Hubbe *et al.* 2017). This can be an advantage during continuous production of a coated paper web, since the evaporation of water from the coated structure will require a longer time to be completed, and the initial immobilization can stabilize the system until drying has been achieved. A related system was reported by Cho *et al.* (2013).

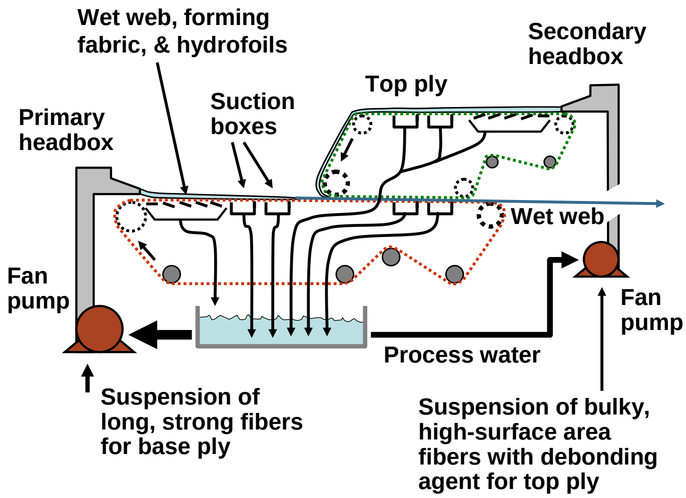

Fig. 34. Simplified schematic diagram of a multi-ply paper forming process, making it possible to employ specialized processing for different layers, *e.g.* for water repellency, stochastic collection of very small particles, and deterministic collection of intermediate-sized particles

Drying Options

Conventional drying

Ordinary drying, for instance on steam-heated rotating cans, is a well-known approach in the industrial production of paper-like materials, including filter papers. The process favors the development of hydrogen bonding among the fibers. Capillary forces, acting at menisci of water in the contact areas between fibers, draw the material together during the evaporation process, leading to an increased apparent density (Page 1993). Such densification will tend to decrease the sizes of pores within the material, and some pores may become closed. In addition, a tendency of the fibrous material to form clumps will decrease the effective surface area, leading to a loss of filtration efficiency (Lee *et al.* 2020).

When papermakers wish to increase the mean pore size and overall void volume of a paper product, the first consideration may be to decrease the energy input for mechanical refining of the pulp (Gharehkhani *et al.* 2015). In the case of kraft pulp, the less-refined fibers will have a greater tendency to retain their three-dimensional cross-sectional shape, leading to a bulkier, less bonded structure of the resulting paper. Another approach is to employ debonding agents during the papermaking process (Garcia *et al.* 2021). Having a structure that is related to that of fabric softeners used in home laundering systems, the debonding agents interfere with the development of hydrogen bonds within and between the parts of the cellulose-based structure during the drying process. Effects of debonding agents on the preparation of air filters were discussed by Garcia *et al.* (2021).

Freeze-drying

The densifying effects of capillary forces can be reduced (but generally not eliminated) by use of freeze-drying methods. As depicted in Fig. 35, freeze-drying is usually implemented by applying vacuum to the material to be dried. In the figure, the wet web of product enters the evacuated zone from the left. Evaporation causes the aqueous solution to freeze. Controlled heat is provided to allow sublimation, while allowing the material to remain frozen. The presence of ice minimizes the effect of capillary forces and allows the surface area of the material to remain high after drying.

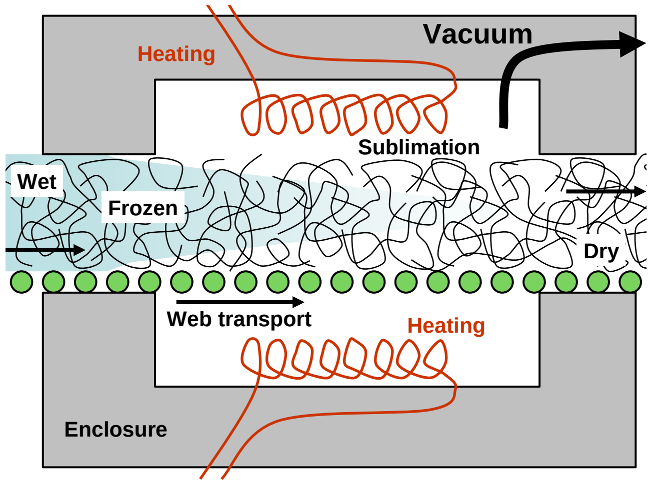

Fig. 35. Schematic of the freeze-drying process, which offers a way to avoid massive loss of specific surface area during the drying of nanofibrillated cellulose and related materials.

Freeze-drying has been employed by various researchers as a way to retain high surface area and a bulky structure during drying of cellulosic material, so as to achieve high-performance filter media. Such research is summarized in Table 4.

Table 4. Studies Using Freeze-drying Methods in the Preparation of Cellulose-based Filter Media

Topics Covered	**Citation**
Beaten softwood and hardwood kraft pulps with partial drying	Mao *et al.* 2008
TEMPO-oxidized nanofibrillated cellulose aerogels for filter media	Nemoto *et al.* 2015
Paper mulberry (kozo) fiber filters; with use of t-butyl alcohol	Yoon *et al.* 2016
Nanofibrillated cellulose having a fibril skeleton morphology	Jimenez-S. *et al.* 2016
Refined softwood kraft fibers in the presence of ethanol, *etc.*	Ma *et al.* 2018b
Fibrillated cellulose in presence of tert-butyl alcohol	Lu *et al.* 2018
Aerogels with wheat straw and konjac glucomannan	Wang *et al.* 2018

Solvent-exchange drying

Toivonen *et al.* (2015) noted the characteristically slow nature of the freeze-drying process and instead used direct drying after replacing water with isopropyl alcohol, followed by exchange to octane. In addition to greatly decreasing the effect of the capillary forces during the subsequent drying, such a solvent exchange process also avoids the formation of hydrogen bonds between cellulosic surfaces in the course of drying. This makes it possible to dry a continuous wet web of filter paper relatively rapidly while maintaining a high specific surface area in the product. In related work, tert-butyl alcohol has been used as the suspending medium when preparing both cellulose-based microporous (Lu *et al.* 2018) and nanoporous filters (Liu *et al.* 2021). The alcohol was found to avoid drawing-together of the cellulose fibrils.

Post-drying Hydrophobization

After a layer of cellulose-based filter media is essentially complete, there can still be an opportunity to adjust the performance attributes of the dry material by various treatments, which often can be carried out in the gas phase (Wulz *et al.* 2021). In particular, such treatments can provide a means to convert hydrophilic surfaces to hydrophobic surfaces. As noted in a recent review article (Szlek *et al.* 2022), gas-phase reactions involving silane chemistry, esterification, and plasma treatments appear to have the best prospects for meeting the needs of product development teams when aiming for hydrophobic cellulose-based media. In principle, a gas phase reaction can be carried out in a counter-current flow system as shown in Fig. 36. Convection and diffusion allow transport of the reagent to the cellulosic surface, making adsorption and reaction possible.

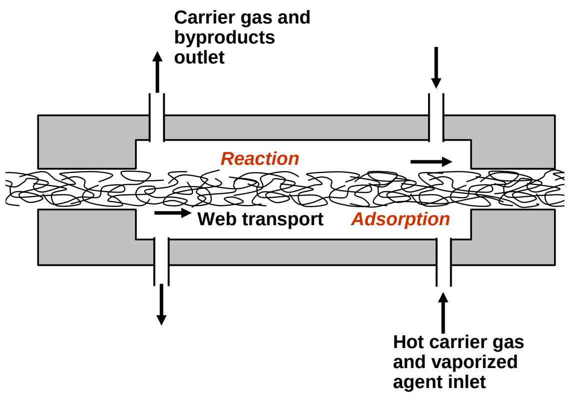

Fig. 36. Concept of a counter-current flow device in which hot carrier gas (to vaporize a reagent of interest) is allowed to pass over the surface of a continuously moving dry web of cellulosic material

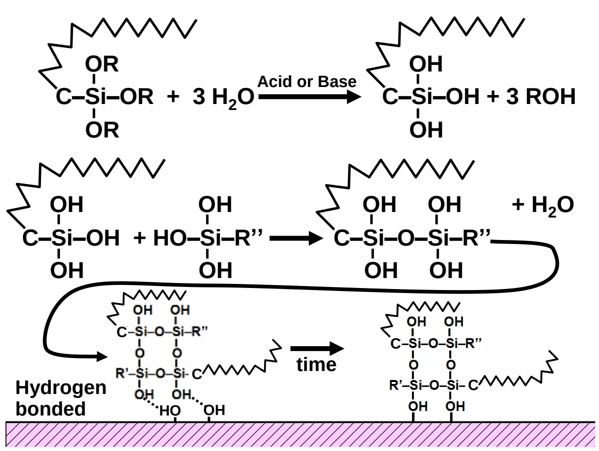

Fig. 37. Main events envisioned during treatment of relatively dry paper with alkyl-trialkoxy silanes plus tetra-ethoxysilane (TEOS) in the presence of equilibrium moisture

Alkyl-trialkoxysilane treatment

Various alkyl trialkoxysilane treatments have been demonstrated in gas-phase reactions, thus providing hydrophobic modification of cellulosic surfaces (Yang and Deng 2008; Yu *et al.* 2019; Shang *et al.* 2021). A tricky feature of such reactions is the fact that a trace amount of water is essential to achieve the desired reaction. The process is outlined in Fig. 37. The amount of moisture present in cellulosic materials at room temperature is likely to be sufficient, but such moisture is subject to evaporation, depending on the temperature and time.

Gas-phase esterification

Among the possible esterification reactions that could be used to render cellulose-based filter media hydrophobic by gas-phase treatments, alkenylsuccinic anhydride (ASA, stearic anhydride, and various long-chain alkyl acid chloride compounds appear to be strong candidates (Szlek *et al.* 2022). The ASA oil most often employed in papermaking operations can be obtained as a waxy liquid, heated sufficiently to cause vaporization (*e.g.* 105 °C), and then transferred to a paper surface using a carrier gas. Gas-phase ASA treatments for cellulosic paper surfaces have been demonstrated in several studies (Zhang *et al.* 2007; Cunha and Gandini 2010a,b; Khoshkava and Kamal 2013). Stearic acid anhydride, which is a solid at room temperature, is less reactive than ASA, but at sufficiently high temperature (say 150 °C) it could be used in a gas-phase treatment of cellulosic materials. By comparison, long-chain alkyl chlorides are highly reactive and can be used for gas-phase reactions (Berlioz *et al.* 2009; Fumagalli *et al.* 2013; Wulz *et al.* 2021). In the cited studies, the treatment temperatures ranged from 160 to 190 °C and the durations were from 2 to 6 h. However, it was not clear from the studies whether or not lower time periods would have been sufficient. No publications were found reporting continuous reel-to-reel gas-phase esterification of paper. A disadvantage of the acid chloride treatments is that HCl, a strong acid, is formed during the reaction. Figure 38 represents an even simpler approach, using a sufficiently high temperature (*e.g.* about 200 °C) sufficient to drive esterification of a common vegetable oil constituent such as oleic acid.

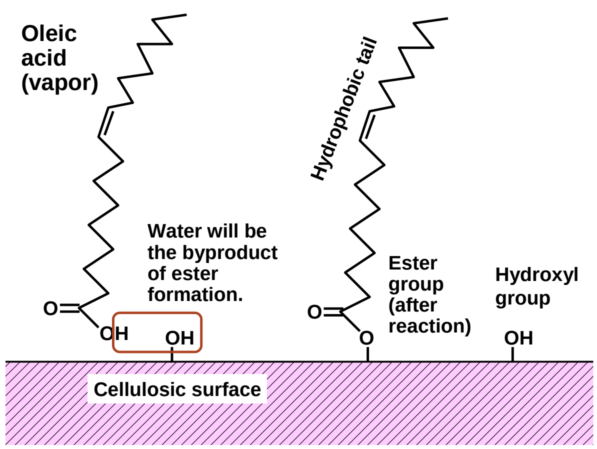

Fig. 38. Schematic depiction of a process of gas-phase esterification of a dry paper web to impart hydrophobicity

CLOSING COMMENTS

Based on the research articles cited in this review, it is clear that cellulosic materials can contribute value in the production of effective media for air filtration. Cellulosic materials offer an advantage over many other materials with respect to coming from renewable plant material, as well as being recyclable and compostable. The costs of many cellulosic materials are favorable in comparison to some of the other materials that they might replace in filter media. Cellulose is a stable molecule that can be expected to meet the durability requirements of many filtration environments. In addition, there are mature technologies for the production and modification of cellulosic materials to meet specific objectives in filter media.

Cellulosic materials often will need to be modified in various ways to meet different requirements for air filtration applications. Technologies are available for the preparation of various different kinds of cellulosic fibers, such as wood-derived cellulose, cotton, and regenerated cellulose products such as rayon. These can be further mechanically refined, as needed to develop increased surface area and/or capacity to form inter-fiber bonds during drying. Freeze-drying methods can be applied if there is a desire to preserve a high surface area, which may be needed to achieve a high capture efficiency of very small particles by the diffusion-interception mechanism. Then, in order to preserve that surface area intact, despite likely moistening of some of the filter media, it will be important to have employed suitable wet-strength and hydrophobic sizing treatments. Antimicrobial treatments also will be important to consider in developing such air filtration products.

In addition to the practical considerations for development of useful air filtration products, the research considered in this review article also point towards a continuing need for further study. From a mechanistic standpoint, it is clear that much has been accomplished in understanding and modeling collections efficiencies and pressure drops, *etc.* However, only a minority of the theoretical work has been focused on cellulosic filter media, which have some specific characteristics. As the world research community continues to place increasing emphasis on the use of plant-based, renewable materials and eco-friendly processing options, it appears that there will be a great amount of needed research in the years ahead related to cellulose-based filter media.

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