



Authenticity Screening for N95 Filtering Facepiece Respirator (FFR) by Field Emission Scanning Electron Microscope (FE-SEM) in Comparison with Particulate Filtration Efficiency (PFE) Analysis

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ABSTRACT

Wearing face masks has become mandatory for many countries around the world as a mean to curb the spread of the SARS-CoV-2 virus transmission. During this difficult time the N95 FFR stock becomes an overnight commodity. The filtration efficiency of the mask is an important concern to protect medical professionals. By description, N95 filtering facepiece respirator (FFR) is a mask that is certified to filter over 95% of the particle size of 0.3 microns. Those numbers are considered to be able to filter out microorganisms and the majority of dust particles and air pollution. Counterfeited FFR's flooded the market and put medical professionals at risk. As the filtration efficiency depends on the physical characteristics and chemical properties of respirator materials, the analysis of fiber topology should shed lights on how to screen counterfeited FFRs from genuine ones. In this study, fiber topology and physical appearances of each filter layer were revealed using low accelerating voltage FE-SEM analysis of four commercial N95 FFR models. The particulate filtration efficiency (PFE) test was operated as means to identify genuine FFRs. The investigation revealed that each layer of N95 FFRs was fabricated in different fiber sizes. The outer and inner layers were larger fibers and with added water repellent capability and the filter in the middle layer was fabricated with smaller fibers. Results from testing of filtration efficiency showed that each mask sample showed a filtration capacity ranging from 94-98% of 0.3 microns particles. Additionally, the SEM image library of authentic fiber images could be used to quickly screen counterfeited FFRs from genuine ones.

INTRODUCTION

Standard N95 masks utilized in health-care settings are better known as filtering facepiece respirator (FFR). In the US, the approval by a National Institute for Occupational Safety and Health (NIOSH), and clearance by the US Food and Drug Administration (FDA) is regulation related to effectiveness to filter air pollution and particles of at least 95% of airborne particles but is not resistant to oil (be rated 'N' based on its level of protection against oil aerosols) [1,2]. To pass the requirement, masks are designed to remove more than 95% of all particles with 0.3 microns or less in diameter. Practically, N95 masks have shown approximately 99.8% particle filtration efficiency of particles of the order of 0.1 microns in diameter [3]. Airborne SARS-CoV-2 virions are of the order of 60-140 nanometers in size. It is known that COVID-19 is also often transmitted through respiratory droplets caused by coughing and sneezing. The diameter of droplet is approximately 5 microns for large droplets and less than 5 microns for smaller droplets, all of which could be filtered by certified N95 FFR [4].

The fiber characteristics and layering of masks and filters directly affect the filtration efficiency. SEM is able to operate in a wide range of magnification and capable of producing high-resolution images with careful sample preparation and control of electron beam parameters which usually could damage delicate fibers [5]. Analysis of biological materials and polymers using SEM requires careful control of beam parameters and conditions, especially if the analysis is carried out on non-coated samples. The accelerating voltages commonly used in SEM image analysis of biomaterials vary between 5 and 20 kV combining with low vacuum to observe fine details on the surface of the structure [5]. The study of Ushiki, et al. indicated that a low (3-5 kV) accelerating voltage is useful for the three-dimensional analysis of the surface morphology of biological non-coated samples and provides a better resolution surface image [7]. High-accelerating voltage is used to obtain more internal structure information [6], however the higher electron accelerating voltages could degrade the polymer fibers in the matter of minutes.

This paper aims to study the fiber physical characteristics of each layer of the N95 filtering facepiece respirators that directly affect the filtration efficiency by using a scanning electron microscope (SEM). The authenticity screening was evaluated with filtration quality and efficiency of FFR samples by measuring particulate filtration efficiency (PFE). SEM conditions were adjusted to appropriate polymer microstructure analysis. Ultimately the library of fiber topology and image is a useful tool to help microscopists worldwide to quickly screen authentic FFRs from counterfeited ones.

METHODOLOGY

Materials

Four models of commercial N95 respirator: A, B, C, and D were selected for testing in this study. B, C and A respirators are dome-shaped model comprised of 3, 4 and 4 layers, respectively. Whereas, D is a three layers foldable model that can flex and expand.

Physical appearance observation

The fiber specimens were prepared for characteristic and topology observation in all mask layers. All four models were dissected into pieces of 1x1 cm² then, each layer was separated as “outer,” “middle1,” “middle2,” and “inner” layers following convention in Figure 1. Field emission scanning electron microscope (FE-SEM) (Quanta 250 Field-Emission SEM, USA) was utilized with secondary electron detector (SED) operating in a low-pressure mode at 5 kV accelerating voltage and the working distance (WD) of 9.7 mm.

Particulate filtration efficiency test

Particulate filtration efficiency (PFE) and pressure drop tests according to ASTM F2299 were performed to evaluate the nonviable

particle retention or filtration efficiency of filter media. N95 FFR samples were tested against particles of 0.3 microns in mean diameter with Gaussian size distribution.

RESULTS AND DISCUSSION

As shown in Figure 2-5. FE-SEM images show well-preserved outline surface of non-coated fiber structures and the complexity of surface morphologies are clearly visible. The layering designs revealed and they were found to be similar in all samples. For example the inner and outer layers of the mask, serve as moisture guard, were fabricated by fibers with a large diameter in range of 20-25 microns. While the middle layer serves as a fine filter, smaller fibers with diameters ranging from 2-5 microns. (Table 1) The pores between the fibers of the inner and outer layers are greater of the order of 50-200 microns which offer breathability while the filtration relies on electrostatic charge to capture the particles microorganisms and virions as they are losing momentum going through layers of fibers.

Particulate filtration efficiency test of N95 FFR samples against 0.3 μm particles are shown in Figure 6. B and C samples presented filtration capacity over 98%, followed by D sample at approximately 97% and 94% for Honeywell sample. The A sample could be a counterfeit FFR as it could not meet the minimum requirement of N95.

According to SEM images, pore sizes of fiber mesh in each layer are greater than 100 microns for all samples. The filtering capability of FFR relies on main 3 mechanisms: 1) inertial impaction where 1 micron or larger aerosol get filtered in the fibrous layer, 2) diffusion where 0.1 microns and smaller undergo Brownian motion around the tortuous porous matrix of the filter fiber, and 3) electrostatic attraction allows the polymer fiber to attract both oppositely charged particles [8] as they slow down through the travel inside the fibrous topology.

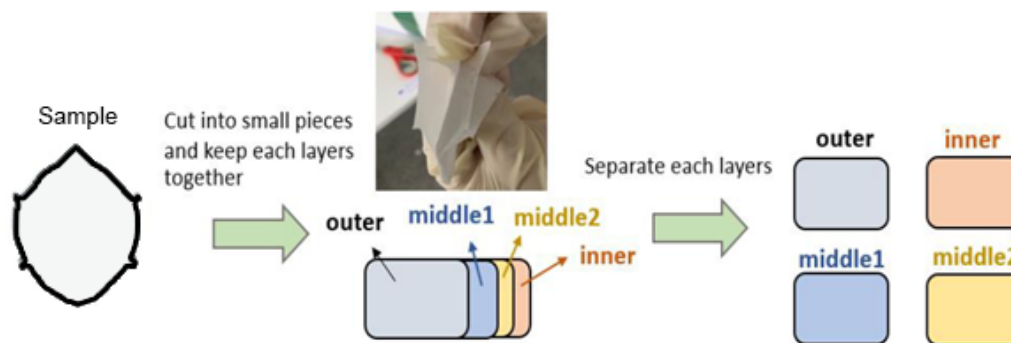


Figure 1. Method of samples preparation and naming convention for SEM operation.

Table 1. Fiber diameter.

sample	Fiber diameter (μm)			
	A	B	C	D
Inner layer	24.21±1.02	25.42±1.53	25.98±2.13	20.17±0.35
Middle1 layer	2.78±0.52	3.13±0.87	45.90±7.06	2.65±0.59
Middle2 layer			4.33±2.58	
Outer layer	23.42±0.55	21.31±0.31	24.92±1.36	20.23±0.58

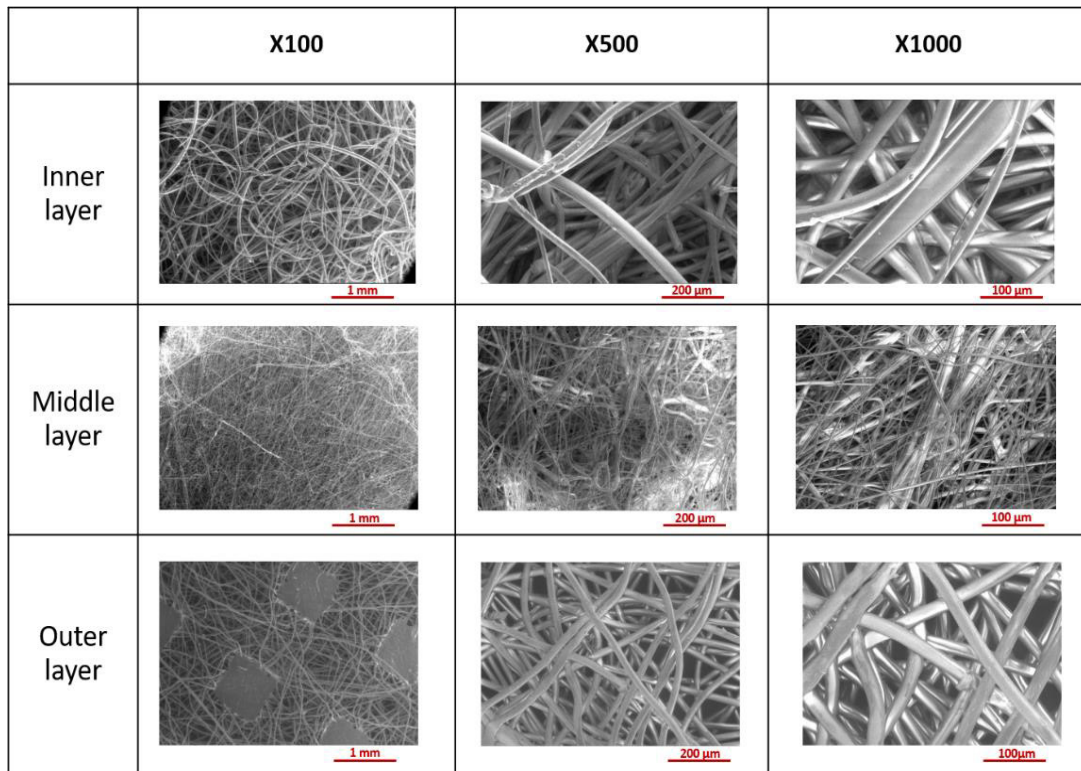


Figure 2. Comparison of the fiber characteristics of each layer of the A mask.

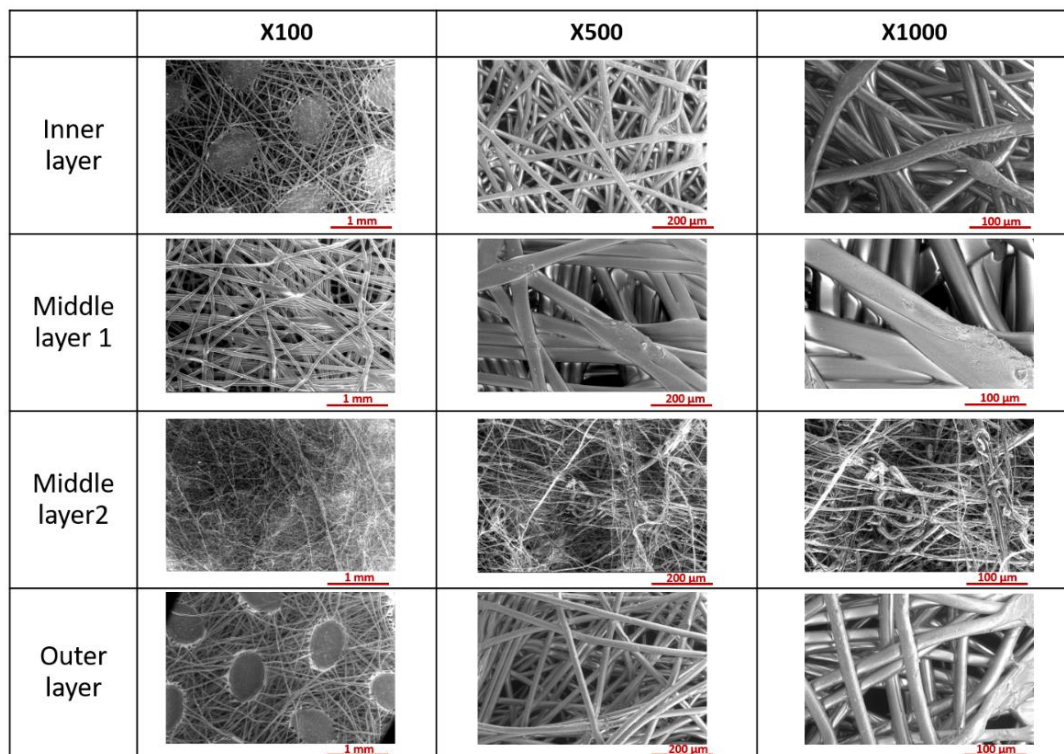


Figure 3. Comparison of the fiber characteristics of each layer of the B mask.

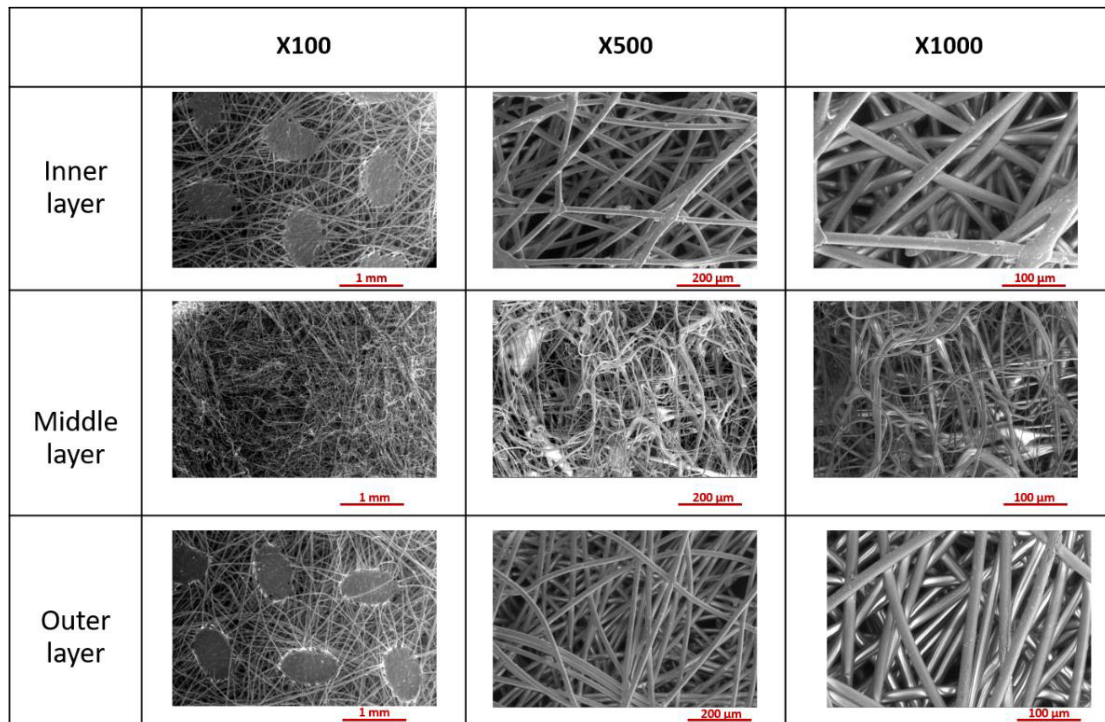


Figure 4. Comparison of the fiber characteristics of each layer of the C mask.

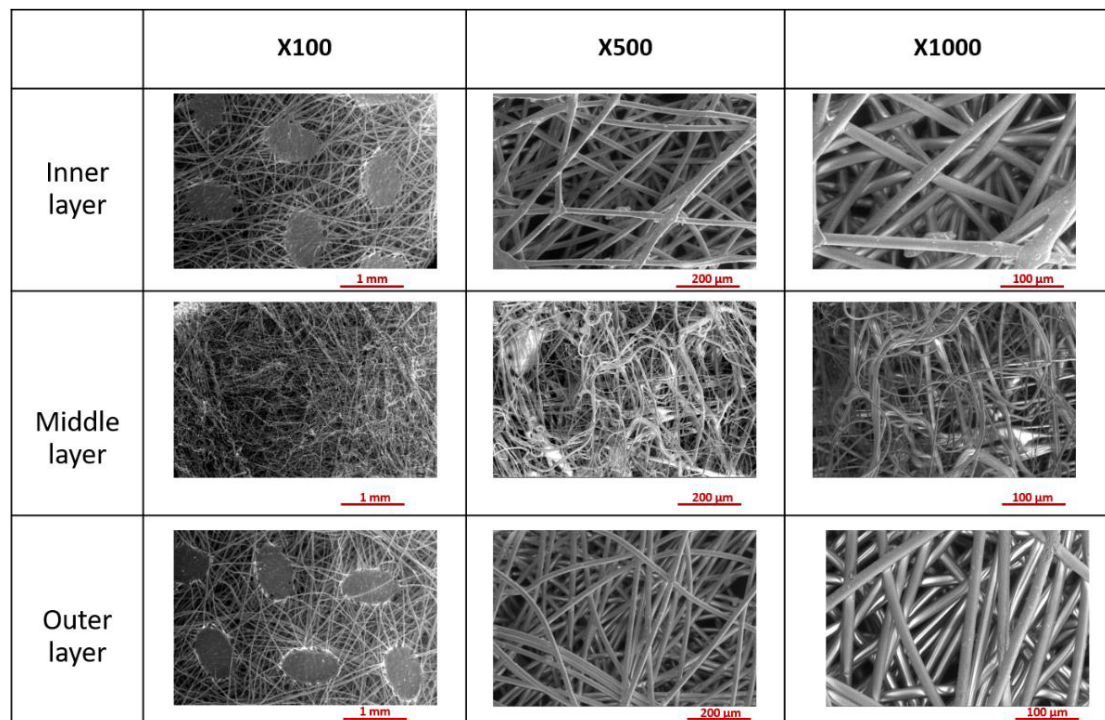


Figure 5. Comparison of the fiber characteristics of each layer of the D mask.

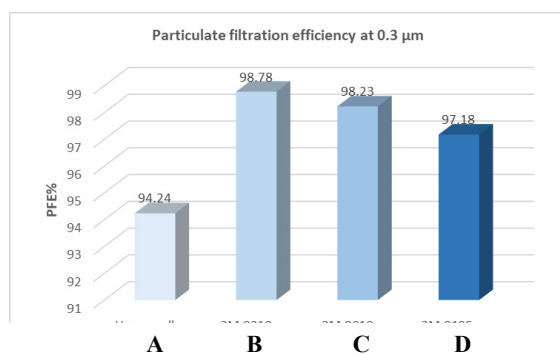


Figure 6. Particulate filtration efficiency (PFE) at 0.3 microns for all FFRs (in new condition).

CONCLUSION

In conclusion, we have presented physical characteristics of fibrous layers to assess the filtration efficiency by SEM analysis of four commercial N95 filtering masks. The results showed that all four mask models had similar designations, i.e., three or more layers of the mask with the inner and outer layers fabricated from large fibers and the middle layer fabricated from finer fibers. For all samples, the pores between the fibers of the inner and outer layer were found to be larger than the pores in the middle layer. The surface characteristics of the fibers in all sample products are visually smooth. In terms of particulate filtration efficiency test, results were found that B, C and D product samples present a filter efficiency of more than 97% which confirm the N95 requirement.

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