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Impact of washing cycles on the performances of face masks

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Abstract

The tension on the supply of surgical and FFP2 masks during the first wave of the COVID-19 pandemic leads to study the potential reuse of these masks. As washing is easily adaptable at home, this treatment solution was retained. In this work, thirty-six references of surgical masks and four FFP2 masks were tested without being worn or washed and after several washing cycles. The results highlighted a great heterogeneity of performances depending on the mask trademarks, both for surgical masks and FFP2. The quality of the meltblown and spunbond layers and the presence/absence of electrostatic charges at the fiber surface are put forward to explain the variability of results, both on differential pressures and filtration efficiencies. The breathability and the particle filtration efficiency of the washed masks were maintained up to 10 washing cycles and met the standard requirements. However, an immersion in water with a detergent induces an efficiency decrease for submicronic particles. This lower performance, constant after the first washing cycle, can be explained by the loss of electrostatic charges during the washing cycle. The modifications of surface properties after washing also lead to a loss of the hydrophobic behavior of type IIR surgical masks, which can therefore no more be considered as resistant to blood projections.

Keywords

Surgical masks, FFP2, Reuse, Washing, Electrostatic charge

Introduction

In the framework of the COVID19 pandemic, both scientific community and the World Health Organization confirmed the major contribution of transport of infectious agent by aerosolized droplets in disease transmission. Transmission of SARS-CoV-2 can occur through direct, indirect, or close contact with infected people through infected secretions such as saliva and respiratory secretions or respiratory droplets expelled by infected contaminated person.

Droplets induced by respiratory activities ($>5-10\ \mu\text{m}$ in diameter) have a propensity to settle quickly (within 1 m from the generation site) because of their size. So contamination through droplet transmission can occur when a person is in close contact (within 1 meter) with an infected person who has respiratory symptoms (e.g. coughing or sneezing; Bourouiba et al., 2014) or who is talking, singing or playing music (He et al., 2021). On the contrary, airborne particles of lower diameters ($< 5\ \mu\text{m}$) can remain in suspension for prolonged periods and expose individuals at a greater distance from the source. Thus, one could inhale aerosols and become infected if the aerosols containing the virus is in sufficient quantity to cause infection within the recipient.

Galton et al. (2010) concluded that particles generated by respiratory activities range from 10 nm to 500 μm . The size of particles emitted by an individual are obviously subject to changes, influenced by the individual himself (mucus properties) (Lee et al., 2019), his activity (normal breathing, talking, sneezing, coughing) and the environmental conditions (temperature, relative humidity). Consequently, the concentration and the particle size distribution that have to be considered when designing barrier mask are very broad. Even if particle size distributions are highly heterogeneous (individual and environment-dependent) their diameter can be assumed as mainly smaller than 5 μm (Papineni & Rosenthal, 1997; Morawska et al., 2009; Johnson et al., 2011; Asadi et al., 2019).

Furthermore, Lindsley et al. (2010) collected cough-generated particles produced by individuals with influenza like symptoms and concluded that viral ribonucleic acid was present in the droplets, whatever their size. Van Doremalen et al. (2020) and Fears et al. (2020) observed that SARS-CoV-2 remained viable and infectious in aerosol after respectively 3 and 16 hours.

As expiratory particles size emitted during breathing and speech are sufficient to carry viable virus (i.e. within 60-140 nm according to several authors (Kim et al., 2020 ; Matsuyama et al., 2020; Park et al., 2020 ; Ren et al. ; 2020), but small enough to be inhaled and penetrate deeper into the respiratory tract (and consequently have more serious health implications) and persist in the air for long time periods, indirect transmission of virus by aerosols might be a plausible hypothesis (Asadi et al., 2020). So, wearing a mask appears essential to limit the pandemic spread as shown by Liang et al. (2020) in their systematic review and meta-analysis. Yan et al. (2019) modelled the evolution of the basic reproduction number R_0 and of the incidence rate, respectively, according to mask efficiency and to the ratio of the population wearing a mask. They concluded that even if masks have a moderate efficiency (around 50%), a negligible transmission occurs if a majority of the population is protected.

Historically, medical face masks are intended for the limitation of the transmission of infective agents during surgical procedures. These masks are used by surgical staff but also by patients and general public for the reduction of contamination during epidemic or pandemic situations.

Before the pandemic, type II surgical masks were recommended in the care services, surgical masks of type IIR for the medical staff during a care with a risk of projection (operating rooms) and filtering facepiece (FFP) respirators during particular care of a patient in special precautions. During the pandemic, filtering facepiece respirators are urged for invasive medical gesture or maneuvers in the respiratory sphere of a patient carrying SARS-CoV-2; type IIR surgical masks for all other types of care. In comparison with FFP, surgical masks are more comfortable, are low cost but fit loosely to the face and are not associated to a protection factor and leakage test. As they present low and highly variable (with filtration velocity and particle size) collection efficiency, surgical masks do not offer protection comparable to filtering facepiece respirators and are considered as a protection for others instead of for oneself.

The tension on the supply of these single-use devices during the first wave of the pandemic of COVID-19 leads to a health use policy that is not without risks for patients and staff. A possible strategy to reduce the mask shortage would be the treatment (to eliminate viral and microbiological risks) and the reuse of these devices. Moreover, during the pandemic, approximately 3.4 billion masks are discharged daily (Benson et al., 2021). This extensive use of face masks, containing polypropylene or other synthetic polymers, induces serious consequences on the environment as these plastic materials may remain in marine or land environments and increase the microplastic and nanoparticle pollutions (Akber et al., 2020; Selvaranian et al., 2021; Sullivan et al., 2021).

Thus, it is interesting to study the feasibility of the reuse of medical face masks and filtering facepiece respirators, both for supply and environmental reasons. After treatments, medical face masks must remain in compliance with EN14863 in terms of filtration efficiency and breathability (Table 1). Concerning equivalence with other international standards, note that a mask meeting the requirements of the American standard ASTM F2100-19 level 1 guarantees compliance with the Type I of the European Standard EN 14683:2019; levels 2 and 3 of ASTM F2100-19 guarantees compliance with the Type IIR of EN 14683:2019. Furthermore, a mask consistent with the Chinese standards YY/T 0969-2013 or YY 0469-2011 meets requirements of the European Standard Type I.

Table 1: Performance requirements for medical face masks (according to EN 14683+AC)

	Type I *	Type II	Type IIR
Bacterial filtration efficiency (BFE)	≥ 95%	≥ 98%	≥ 98%
Differential pressure	< 40 Pa/cm ²	< 40 Pa/cm ²	< 60 Pa/cm ²
Splash resistance pressure	Not required	Not required	≥ 16 kPa
Microbial cleanliness	≤ 30 cfu/g	≤ 30 cfu/g	≤ 30 cfu/g
* Type I medical face masks should only be used by patients and other persons to reduce the risk of spread of infections particularly in epidemic or pandemic situations. Type I masks are not intended for use by healthcare professionals in an operating room or in other medical settings with similar requirements			

Regarding filtering facepiece respirators, they should preserve the following requirements after washing cycles (table 2). It should be mentioned that the filtration efficiency is determined on the material constituting the mask, as for surgical masks, and that leakage tests are also carried out on the filtering facepiece respirators worn on the face.

Table 2: Performance requirements for filtering facepiece respirators

	N95 (USA)	FFP2 (Europe)	KN95 (China)
Norm	NIOSH-42C-FR84	EN 149-2009	GB2626-2006
Filtration efficiency	≥ 95%	≥ 94%	≥ 95%
Test aerosol	NaCl	NaCl, Paraffin oil	NaCl
Pressure drop (Pa)	≤ 343 Pa (at 85 l/min)	≤ 70 Pa (at 30 l/min) ≤ 240 Pa (at 95 l/min)	≤ 350 Pa (at 85 l/min)

From the beginning of the pandemic, the decontamination and the reuse of N95 filtering facemask respirators have been the subject of an increasing number of studies (Schumm et al., 2021). As they preserve N95 mask integrity in terms on penetration, air flow resistance and physical appearance, the most promising methods seem to be UV irradiation (Viscusi et al., 2007; Bergman et al., 2010; Liao et al., 2020), ethylene oxide (Viscusi et al., 2009; Bergman et al., 2010) and vaporized hydrogen peroxide decontaminations (Viscusi et al., 2009; Bergman et al., 2010, Fisher et al., 2020; Richter et al., 2016, Cai and Floyd, 2020). Regarding heat treatment, Liao et al. (2020) showed that heating (dry or in the presence of humidity) at temperatures up to 100°C can preserve the efficiency of the mask after 10 cycles whereas Fisher et al. (2020) highlight that 70°C dry heating allows maintaining performances only up to 2 cycles. However, the conservation of performances not only depends on the kind of treatment but also of the N95 model (Rodriguez-Martinez et al., 2020). Microwave oven use (Viscusi et al., 2011; Bergman et al., 2010) and bleach treatment (Viscusi et al., 2007; Bergman et al., 2010) do not induce significant changes in penetration and air flow resistance but lead to media or head straps melting on some masks after a few minutes and tarnishing of nosebands, respectively. Treatment with an autoclave, immersion in a 70% isopropyl alcohol (Viscusi et al., 2007) or ethanol (Liao et al., 2020) conduct to a drastic degradation of filtration performances which are no more consistent with normative criteria. In the same way, when testing with a polydisperse sodium chloride with a count mean diameter of 40 nm, Suen et al. (2020) concluded that, of the different treatments on surgical masks, non-fluid-based methods such as UV irradiation maintain filtration efficiency after three cycles while immersion in water or alcohol induces the loss of electrostatic charges of the mask. To recover electret effect of masks and increase the efficiency degraded by sterilization treatments, Hossain et al. (2020) proposed a simple recharging method based on an electrical field. Similarly, Wang et al. (2020) showed that after hot water decontamination, the drying with a hair drier allows recovering 90% of the electret effect of masks.

As washing can easily be realized at home, this treatment solution was retained and the influence of washing cycles on the performances of surgical masks and filtering facepiece respirators was studied. It should be noticed that disinfection performances (i.e. elimination of viral and microbiological risks) were not addressed in the paper. However, washing procedures applied lead to a total number of colony forming units lower to the normative limit (30 cfu/g) described in the EN 14683+AC standard.

Materials & Methods

Samples

Thirty-six references of surgical masks (5 masks of type I, 13 type II and 18 type IIR) and four FFP2 masks were tested without being worn or washed (considered as new) and/or after some washing cycles (Appendix 1). For references appearing several times in the table, different batches were tested over different periods of time to evaluate the repeatability.

Washing procedure

Depending on the origin of the washed masks, various protocols were applied:

- W1: a cycle in an industrial washer machine, which here corresponds to 12 min of washing at 60°C with 5 mL/kg of disinfectant and 1 mL/kg of detergent; 1 min of draining, 3 min of rinse at 30°C and 3 min of spin at 550 rpm. The masks were then placed in the dryer with 3 cycles (3 min drying / 3 min cooling) gradually increasing the temperature up to 45°C and decreasing it to 20°C.
- W2: a washing cycle corresponds to the one used for the gown washing in the teaching hospital of Nancy; i.e. 15 min of washing at 60°C with a detergent, 15 min of washing at 60°C with a bleaching agent, 2 min of intermediate spin, 3 min of rinse and 3 min of fast spin.
- W3: Masks were washed in an individual washer machine at 40°C and with a liquid detergent. After a rinse and an intermediate spin at 500 rpm, the masks were dried in open air.
- W4: The washing was realized at 60°C with a detergent during 30 minutes. After 4 cycles of rinse (3 min, 3 min, 2 min, 2 min) and a 5-min spin at 800 rpm, the masks were placed in a dryer during 40 min at 80°C.
- W5: Masks were washed in an individual washer machine at 30°C with or without a liquid detergent.

Breathability and particle filtration efficiency

As mentioned in the NF EN 14683 standard “Surgical masks - Requirements and test method”, test specimens are cut from complete masks. These samples are taken far enough away from the bonding areas. All the layers composing a medical face mask sample are placed in a filter holder with a filtration surface of 28.3 cm². This surface is smaller than the one recommended for BFE in the standard but sufficient to be representative of the nonwoven media.

The value of the initial pressure drop is recorded for various air flow rates (between 10 and 50 liters per minute). In a laminar regime, the permeability can be deduced from the slope of the straight line representing the pressure drop as a function of air flow rate. Knowing the surface filtration, the breathability of the medical face mask, corresponding to the differential pressure per surface area, is calculated.

According to the EN 14863 protocol, a suspension of *Staphylococcus aureus* should be nebulized and the particles generated (with a mean size of $3 \pm 0.3 \mu\text{m}$) should be collected on a six-stage cascade impactor. The bacterial filtration efficiency is determined by counting the number of colony forming units on all the plates, after an incubation at 37°C during 20 to 52 hours. The experimental procedure was adapted and a Particle Filtration Efficiency (PFE) was determined instead of a Bacterial Filtration Efficiency (BFE).

Moreover, Stadnytski et al. (2020) demonstrated that the probability that a 50 μm -diameter droplet generated from a homogeneous distribution of oral fluid contaminated by COVID-19, prior to dehydration, contains at least one virion is close to 37%. For a 10- μm droplet, this probability drops to 0.37%, and the probability that it contains more than one virion is negligible. But depending of environment conditions, these large droplets containing virus can evaporate and consequently become smaller, remain in suspension and expose individuals. In this study, the mask efficiency is determined not only for 3- μm particles but also for submicron particles. The size range of the virus (60-140 nm) could then be assumed as a hypothetical lower size limit.

For the determination of surgical mask efficiency, a micron-sized DEHS (di-ethyl-hexyl-sebacate) or a submicron salt (KCl) aerosol was produced by an AGK 2000 Palas[®] generator and diluted with compressed air; while, a NaCl aerosol was generated for the measurement of FFP2 efficiency. For both kinds of masks, filtration velocity is adjusted at 9.6 cm/s, (corresponding to the velocity used in the EN 14683+AC standard) and the particle size distribution is measured upstream and downstream of the filter with various detectors (size spectrometer or photometer), depending of the nature of the aerosol (cf. table 3). The DEHS mean number aerodynamic equivalent diameter (APS measurement) was close to 0.85 μm and the KCl and NaCl mean number mobility equivalent diameters (SMPS measurement) were close to 85 nm (corresponding to a mean mass diameter close to 600 nm).

Table 3: Detectors and test aerosols used according to mask type

	Test aerosol		
	DEHS	KCl	NaCl
Aerodynamic Particle Sizer	Surgical masks		FFP2
Scanning Mobility Particle Sizer		Surgical masks	FFP2
Flame photometer			FFP2

The fractional efficiency was calculated as follows for a given particle size, d_p :

$$E_N(d_p) = 1 - \frac{C_{N,down}(d_p)}{C_{N,up}(d_p)} \quad (1)$$

where $C_{N,down}$ and $C_{N,up}$ were the particle number concentration downstream and upstream of the filter, respectively. In addition to fractional efficiency, overall filtration efficiency, based on NaCl mass concentration measurements, was determined specifically for FFP2 mask according to flame photometer in agreement with EN149+A1.

For surgical masks, efficiency measurements were repeated 3 times on the same mask sample which corresponds to a repeatability analysis. Moreover, measurements were conducted on at least three samples of a same medical face mask (reproducibility test).

Surgical masks and filtering facepiece respirators performances are determined on the LRGP and IRSN test benches, respectively. More details on experimental test benches are available in a previous paper (Bourrous et al., 2021) which demonstrated that despite different test aerosols, measurement methods, protocols and test bench configurations, permeability and collection efficiency for 3 μm particle diameter are in good agreement.

According the EN 14683+AC protocol, each sample of surgical mask shall be conditioned at $(21 \pm 5) ^\circ\text{C}$ and $(85 \pm 5) \%$ relative humidity for a minimum of 4 hours to ensure equilibrium

prior to testing. Preliminary tests with and without this conditioning procedure lead to similar results, in terms of permeability and collection efficiency. To simplify our test procedure, this conditioning step was consequently not applied, for both surgical and FFP2 masks.

Projection resistance

The experimental procedure and equipment needed to determine the resistance against penetration by synthetic blood are described by ISO 22609:2004 standard. In the health-care context, the experimental set-up described in the standard was slightly adapted to the apparatuses and materials available. The pneumatic valve was replaced by an electrovalve SMC VX21 and a needle (gauge 18) was used as a canula. It should be stressed that the properties of the valve assembly were consistent with those of the standard: i.e., 13 mm long canula (instead of 12.7 mm), an inner diameter of 0.8 mm (instead of 0.84 mm) and the possibility to adjust the injection duration by 0.1 s step. The set-up was placed in a glove box which can be opened rapidly in order to check the blood stains on the mask placed on a holding fixture (Figure 1). A targeting plate with a 0.5 cm hole is located 1 cm in front of the mask and cups are used to collect the blood in excess. Before testing the washed masks, calibration and validation tests were performed on new masks.

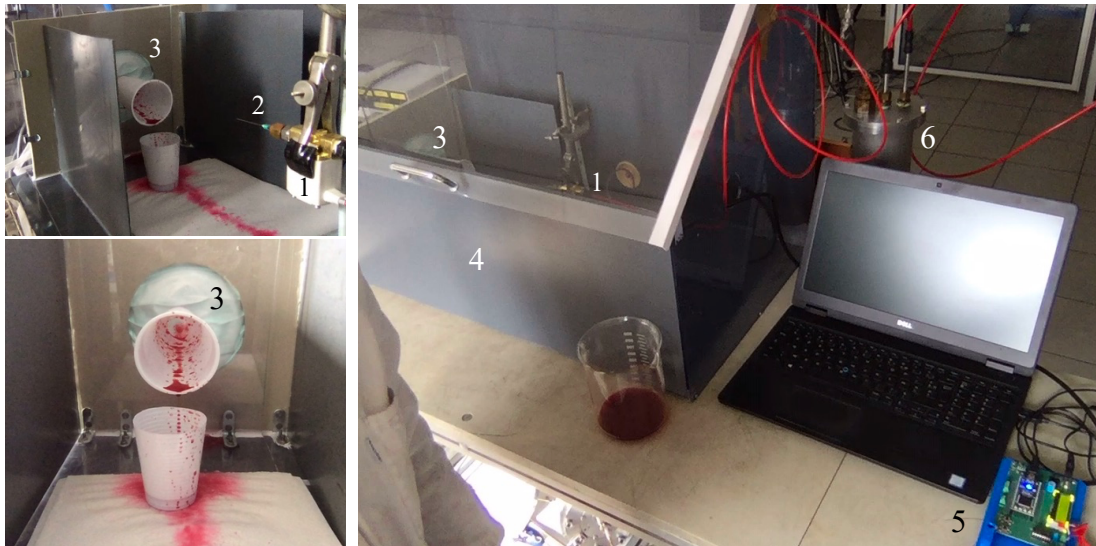


Figure 1: Experimental setup for projection resistance tests (adapted from ISO 22609:2004)

(1: Electrovalve; 2: Needle; 3: Mask holding fixture; 4: Glove box; 5: Valve controller; 6: Synthetic blood tank with pressure gauge)

However, due to the lack of some reagents (urgency of the situation combined to shortage due to the lockdown), an alternative but approaching mixture was tested.

The preparation and composition of the synthetic blood is detailed in Annex B of the ISO 22609:2004 standard. In addition to distilled water, a thickening agent and red colorant are the products used adjust the viscosity, surface tension and color of the synthetic blood. However, as this study was both urgent and carried out in a context of shortage due to the confinement, an alternative blood composition was developed (Table 4).

As different reagents were used, it was essential to check and adjust the synthetic blood viscosity and surface tension. The surface tension was determined using the stalagmometric method (Tate's method). By weighting a single droplet (m) dropping from a canula of known radius (r), the surface tension γ can be determine after several replicates:

$$\gamma = \frac{m \cdot g}{2 \cdot \pi \cdot r} \quad (3)$$

After addition of a small amount of surfactant, the surface tension of the synthetic blood is 40 ± 2 mN/m, which is consistent with the expected (42 mN/m). Correlation of the literature was used to determine the dynamic viscosity of glycerol/water mixtures and reach 4 mPa.s, which corresponds to the viscosity of blood at 37°C (Cheng, 2008).

Table 4: Comparison of the synthetic blood compositions

Reagents	Standard	Alternative mixture
Water	500 mL	500 mL
Thickening agent	12.5 g Ammonium salt of acid-acrylic	12.5 g Sodium salt of acid-acrylic + 150 mL glycerine
Colorant	5 g Direct Red 81	3 g Direct Red 28
Surfactant	-	0.3 g Tween

Preliminary tests were carried out at various pressure and injection time. In order to obtain the same volume generated in 0.57 s (here 0.6 s) at 21.3 kPa (standard values), the pressure has to be slightly increased up to 29 kPa, which can be due to different pressure drops in the valve assembly. Under these conditions, the blood volume injected during a test agrees with the volume imposed by the ISO standard.

Results & Discussion

Breathability and particle filtration efficiency of new masks

Performances of each surgical mask trademark are represented on figures 2 and 3. Horizontal dashed lines correspond to standard requirements. A cross means that the considered masks do not reached the recommendations ($> x$ % for efficiency and $< x$ Pa/cm² for differential pressure). Error bars correspond to standard deviations determined from a reproducibility analysis on 4 to 8 samples for differential pressure and on 2 to 4 samples for collection efficiency for 3 μm particle diameter. These measurements conducted on masks of a same batch and/or on two samples cut in a same mask provide an indication on the heterogeneities of this non-woven material.

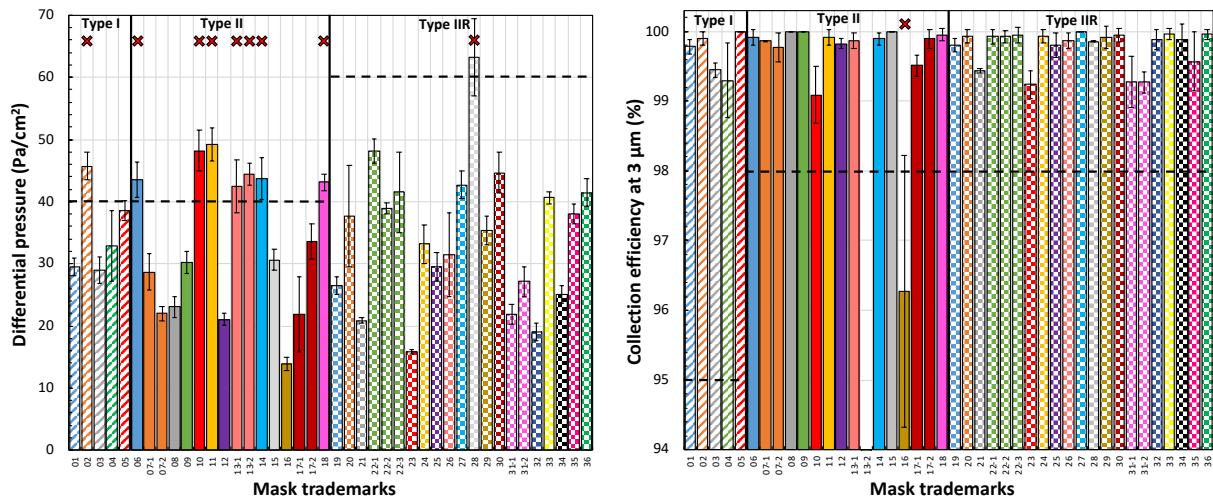


Figure 2: Differential pressure of the various mask trademarks

Figure 3: Collection efficiency of the various mask trademarks

Some surgical masks are not sufficiently breathable and not in compliance with EN 14683+AC:2019 standard. It should be pointed out that the protocol does not fulfil the normative requirements. Nevertheless, without corresponding exactly to the conditions of the standard, the tests carried out allow a precise intercomparison of the different masks. Despite similar structure (spunbond / meltblown / spunbond), the mask trademarks of a same type present strong heterogeneities in terms of differential pressure. Despite a particle filtration efficiency very high (>99%), even for type I masks, this parameter seems to be trademark-dependent.

Only the surgical mask presenting the lower differential pressure (Ref. 16) does not meet the requirements of the European Standard EN 14683:2019 in terms of collection efficiency. For this trademark, the collection efficiency measurements realized on 3 samples are highly heterogeneous (98.5%, 95.1% and 95.2%). Tests carried out with a DEHS aerosol could therefore be considered as an alternative to bacterial filtration efficiency measurements for which the uncertainties are numerous (Pourchez et al., 2021).

A Mann-Whitney U statistical test is carried out in order to conclude with performances of the different kinds of masks. This non-parametric test is used to determine if all the values from two groups are independent of each other. Even if they give very similar results, for distributions sufficiently far from Gaussian, the Mann-Whitney U test is considerably more efficient than the Student one.

It consists in assigning numeric ranks (by ascending order) to all the observations (permeability or efficiency in our case) of the two groups and then determining the sum of the ranks, S , of each group. A statistic, called U , is calculated for each group:

$$U_1 = S_1 - \frac{n_1 \cdot (n_1 + 1)}{2}$$
$$U_2 = S_2 - \frac{n_2 \cdot (n_2 + 1)}{2}$$

Where, n_1 and n_2 represent the sample size of the groups 1 and 2, respectively. S_1 and S_2 are the sum of the ranks of each group. The mean, $M(U)$, and the variance, $V(U)$, are determined:

$$M(U) = \frac{1}{2} \cdot n_1 \cdot n_2$$
$$V(U) = \frac{1}{12} \cdot (n_1 + n_2 + 1) \cdot n_1 \cdot n_2$$

The experimental standardized value, Z is calculated, considering a continuity correction for small groups:

$$Z = \frac{|\min(U_1; U_2) - M(U)| - 0.5}{\sqrt{V(U)}}$$

With a confidence interval of $(1-\alpha)$ for a two-sided test, the values of breathability and particle filtration efficiency for the population 1 and the population 2 are considered to be the same, if the absolute value of the experimental standardized value, Z , is comprised between 0 and the t-value of the Student distribution corresponding to $(1-\alpha/2)$ and (n_1+n_2-2) degrees of freedom. For the test conducted on the filtration efficiency, the sizes of the population are 13,

44 and 72, respectively for surgical masks of type I, II and IIR; while for the test on the breathability, the populations contain 24, 89 and 127 values, respectively for types I, II and IIR.

The differences between the populations of Type I, II, IIR masks have been statistically tested in pairs and, among all the references tested, there is no significant variation of breathability and collection efficiency for 3 μm particle diameter between the different kinds of masks for a confidence interval of 95%. The observations (breathability and efficiency) can be considered as similar until a limit significance level (table 5). Each percentage indicates the maximal risk of being wrong supposing that the performances of masks of various types are similar.

Table 5: Confidence interval obtained with Mann-Whitney U statistical test

	Type I vs Type II	Type I vs Type IIR	Type II vs Type IIR
Breathability (degree of freedom)	67.7% (111)	73.2% (149)	27.0% (214)
Particle Filtration Efficiency (degree of freedom)	83.2% (55)	80.5% (83)	6.4% (114)

Even if the performances do not seem to be influenced by the kind of surgical masks, results of table 5 tend to highlight that the similarity is higher between types II and IIR, both for the breathability and for the efficiency for 3 μm particle diameter.

Filtering facepiece respirators (FFP2) should collect more than 94% of a NaCl aerosol with a mean mass diameter close to 600 nm. For the four FFP2 tested, the total particle efficiency determined with a photometer was higher than 99.5% (figure 4). Calculating this total collection efficiency from the SMPS upstream and downstream concentration conducted to lower values. Indeed, particles with a mobility-equivalent diameter higher than 550 nm were not counted by the SMPS while they were collected with a high efficiency due to interception and inertial mechanisms.

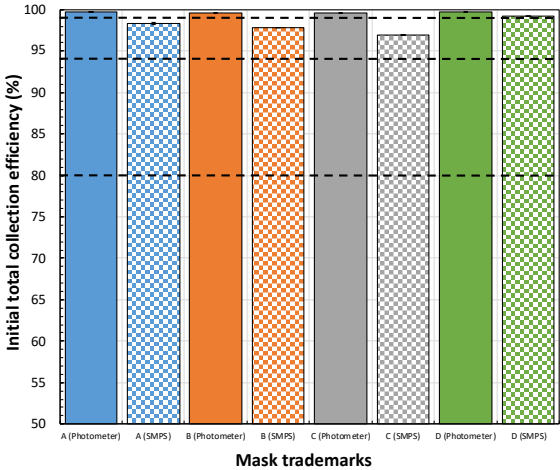


Figure 4: Total collection efficiency of filtering facepiece respirators

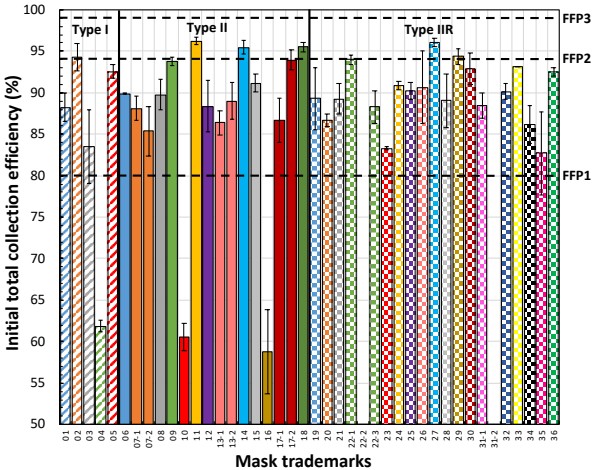


Figure 5: Total collection efficiency of surgical masks

This SMPS total collection efficiency was also determined for the surgical masks (figure 5). As expected, efficiencies of filtering facepiece respirators are higher due to their structure (higher solid volume fraction and/or number of layers), but most of the surgical masks present

performances similar to those of FFP1 and some of them could be considered as efficient as FFP2, in regards to the EN 149-2009 standard.

It should be reminded that only the material constituting masks are tested and that leakage are not considered. Three references (04, 10 and 16) have a total collection efficiency close to 60% and the determination of spectral efficiency will allow giving some explanations on these lower performances.

Figures 6 to 9 represent the collection efficiency of the different kinds of masks according to the particle diameter. As particle concentrations upstream and downstream of a mask sample were measured with a SMPS and an APS, the diameter on the abscissa axis is a mobility-equivalent diameter on the range 20-500 nm and an aerodynamic diameter for particles higher than 1 μm . It should be noted that despite various measurement principles and equivalent diameters, the instrumental responses are in reasonably good agreement. As surgical masks are constituted of non-woven material, their spectral efficiency present a classical U-shape due to the interaction of the main collection mechanisms (diffusion, interception, inertial impaction and electrostatic effect) and a most penetrating particle size (MPPS) between 0.2 and 0.5 μm . For filtering facepiece respirators, this MPPS is shifted to lower diameters due to electrostatic effects. These results also highlight a great heterogeneity of performances depending on the mask trademarks, both for surgical masks (whatever the type) and FFP2. As the collection efficiency and the width of the MPPS are directly dependent of the fibrous structure (solid volume fraction, fiber size distribution, thickness), the quality of the meltblown and spunbond layers can probably contribute to these heterogeneities. If the collection efficiency for surgical masks is, in most cases, higher than 70-80% for the whole range of particle diameters, some references (04, 10 and 16) present efficiency lower than 40% for the most penetrating particle size. This marked evolution is probably due to the absence of electrostatic charges at the fiber surface of these surgical masks.

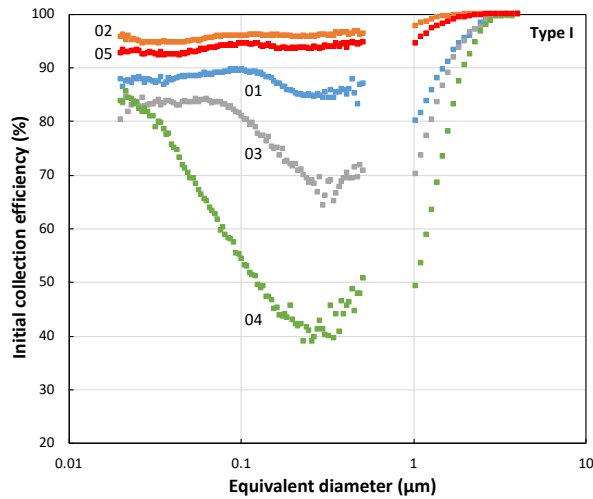


Figure 6: Spectral efficiency of type I masks

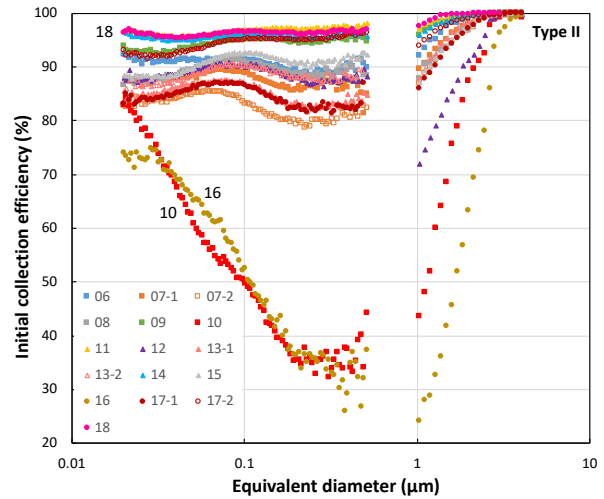


Figure 7: Spectral efficiency of type II masks

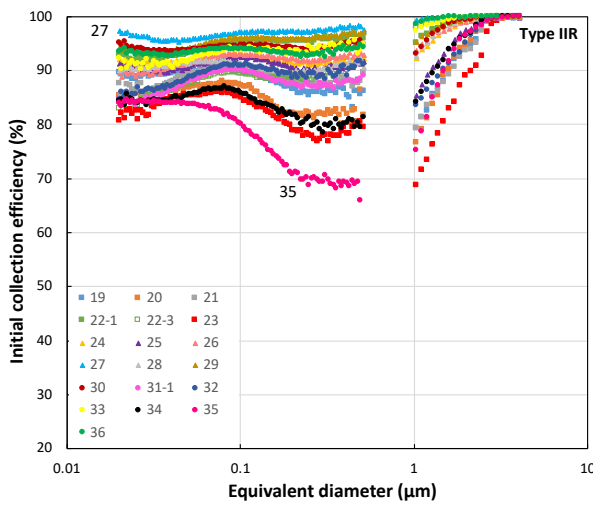


Figure 8: Spectral efficiency of type IIR masks

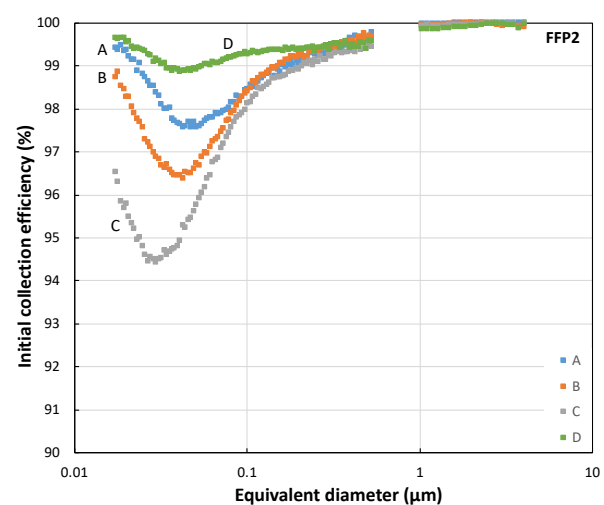


Figure 9: Spectral efficiency of FFP2 masks

Breathability and particle filtration efficiency of washed masks

The performances of washed surgical mask trademarks are represented on figures 10 to 13. As previously, the horizontal dashed lines correspond to standard requirements. The error bars correspond to standard deviations determined from a reproducibility analysis on 4 to 8 samples for differential pressure and on 6 to 12 samples for collection efficiency for 3 μm particle diameter.

Whatever the reference and the washing procedure, the first cycle induces a slight decrease of differential pressure. Therefore, if masks meet the requirements of the standard before washing, they also remain in compliance with it after a washing cycle. As there is a relationship between the breathability and the collection efficiency (Bourrous et al., 2021), this modification of the non-woven structure leads to a decrease of collection efficiency for 3 μm particle diameter. However, performances remain constant hereafter a cycle and up to 10 cycles, i.e. the maximal cycle number tested for 7 references of surgical masks.

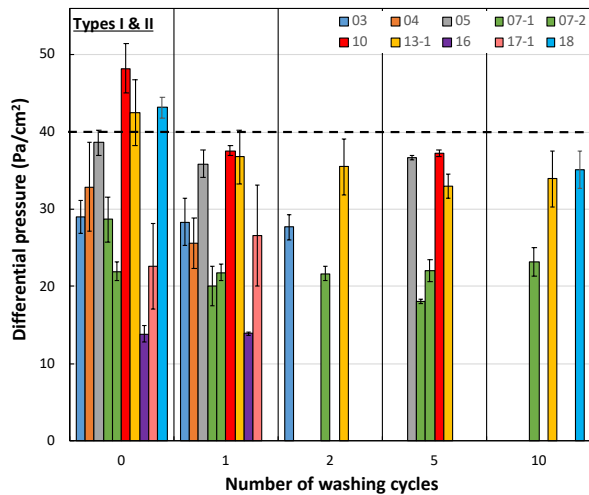


Figure 10: Differential pressure according to the number of washings (type I and II masks)

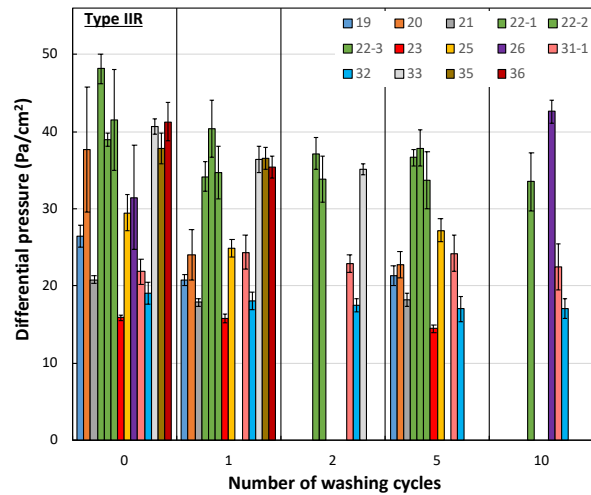


Figure 11: Differential pressure according to the number of washings (type IIR masks)

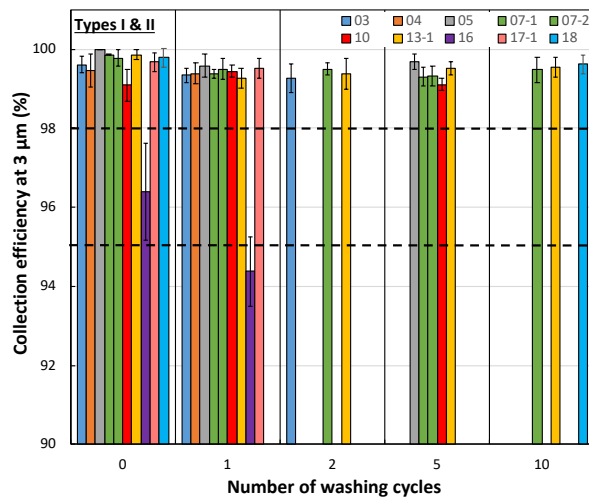


Figure 12: Collection efficiency according to the number of washings (type I and II masks)

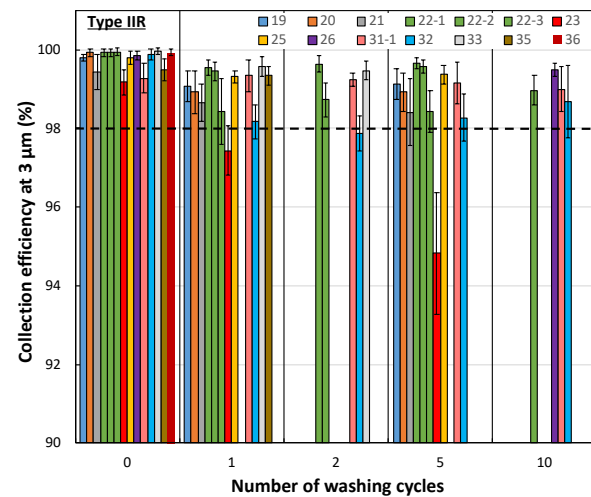


Figure 13: Collection efficiency according to the number of washings (type IIR masks)

For each reference, the Mann-Whitney U test is carried out to determine if washing is statistically responsible for a performance decrease (with a significance level of 5%). This statistical test shows that, for both the breathability and the efficiency for 3 μm particle diameter, the results obtained on new masks and washed masks cannot be considered significantly different, except for some trademarks. A significant modification of breathability is noted for the references 10 and 13-1 after 5 washing cycles but it does not lead to a significant decrease of collection efficiency. A significant decrease of efficiency can also be observed after the washing of the references 16 and 23. Nevertheless, trademarks 10 and 16 have been previously identified as less efficient and highly heterogeneous, more samples should be tested to definitively conclude on the influence of washing on these surgical masks. Concerning the reference 23, the inner and outer layers of the mask have the particularity of being composed of cellulose fibers. This characteristic could maybe explain the PFE decrease which is not significant for the majority of the masks composed of polypropylene fibers.

If the collection efficiency for 3 μm particle diameter remains greater than 95 or 98% whatever the type of surgical mask and the number of washings, the performance of the masks is

impacted by the washing for lower particle sizes (Figures 14 to 17). As the presence of an intermediate meltblown layer of charged polypropylene fibers contributes to the collection efficiency by electrostatic effects, this decrease in efficiency, constant after a cycle, can be explained by the loss of electrostatic charges during the washing cycle as confirmed by the results obtained on a mask discharged by immersion in isopropanol (Figure 16). A Kelvin probe was used to measure the global charge of polypropylene fibers for one of the filtering facepiece respirators (reference A). The registered mean surface potential, close to -500 V for the non-washed FFP2, decreases until a value close to -20 V after a washing cycle and confirms the charge neutralization and the removal of the electret effect on the washed masks. Moreover, the experiments conducted on the reference 16, with an efficiency lower than 40% for the most penetrating particle size before washing (Figure 7), confirm the absence of electrostatic charges at the fiber surface; the collection efficiency before and after washing being similar on the whole particle size range.

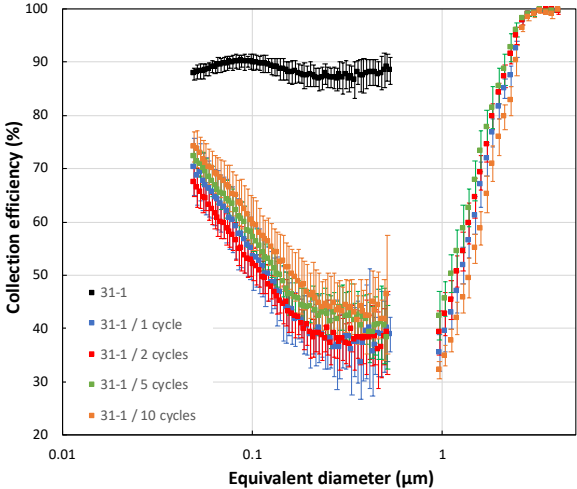


Figure 14: Spectral efficiency of washed surgical masks (reference 31)

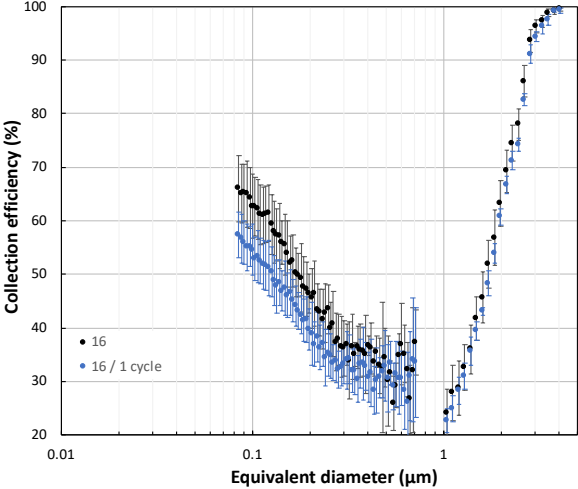


Figure 15: Spectral efficiency of washed surgical masks (reference 16)

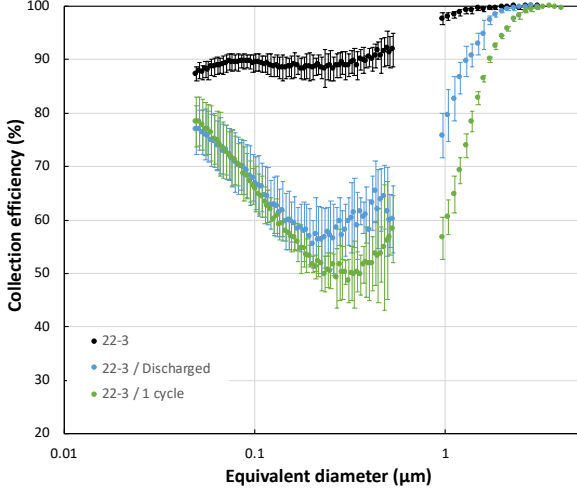


Figure 16: Spectral efficiency of washed and discharged surgical masks (reference 22)

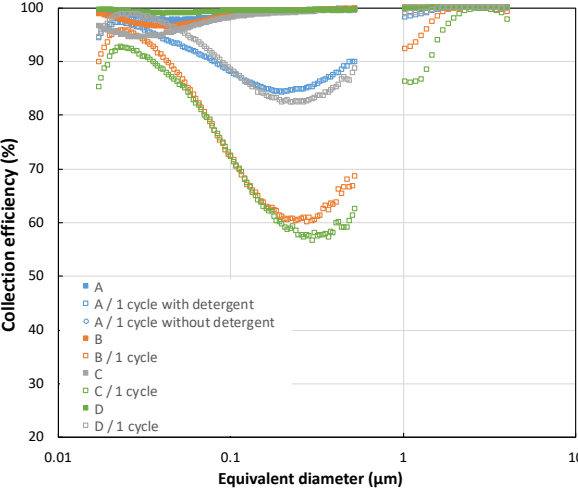


Figure 17: Spectral efficiency of FFP2 masks washed with or without detergent

As a washing without detergent maintained the performances of the mask sample at the same level as the new FFP2 (Figure 17), the loss of electrostatic effects could likely be attributed to

the presence of cationic surfactants in fabric softeners. These compounds, notably esterquats, possess excellent antistatic properties and are used to prevent the accumulation of static charges and make the textile surface more conductive (Murphy, 2015; Mishra and Tyagi, 2007). This positive charge of cationic surfactants (Agarwal et al., 2012) reinforce results obtained with the Kelvin probe. Visualizations of one of the filtering facepiece respirators (reference A) with a JSM-7900F (Jeol) scanning electron microscope (SEM) as well as analysis by X-ray energy-dispersive spectrometry (EDX) suggest that surfactant residues (presenting significant contributions if Fe, Mg, Al and Si) and an organic film (mainly C, O and N) could be deposited at the fiber surface (without modification of their diameter or abrasion of their surface) after a washing cycle and contributes to their neutralization (Figure 18). Such observations have also been highlighted by Parvinzadeh and Hajiraissi (2008) and Obendorf et al. (2009).

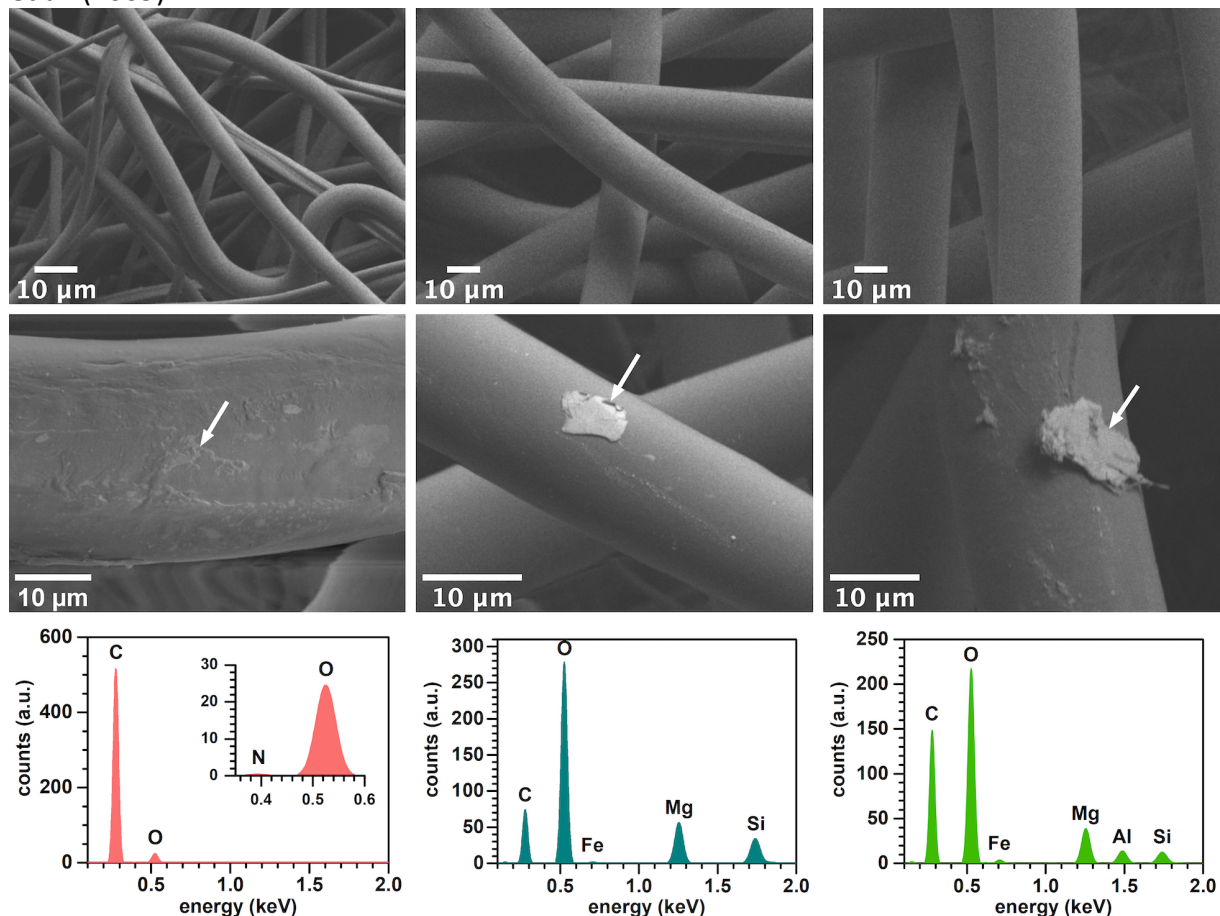


Figure 18: SEM images of filter fibers before washing (top row) and after washing with detergent (middle row). The bottom row shows EDX spectra of impurities deposited on the washed fibers, indicated by the arrows in the SEM images

Projection resistance

The projection resistance tests were performed on IIR masks under the conditions described by the standard, at a blood ejection rate of 550 cm/s corresponding to a blood pressure of 16 kPa. The tests were repeated once for each type of mask under the same conditions. To be fully compliant with ISO 22609, nearly 30 tests should be performed for each type of mask, which was obviously not possible in the pandemic and lockdown context.

Visual observations show that, after one to two washings, the 'anti-splash' properties of the masks are preserved according to the ISO 22609 standard, i.e. no trace of blood was detected on the inner face of the mask, 10 seconds after the blood projection. However, the protective properties of the first layer are degraded after about 4 washings and the blood enters the mask. As the blood only accumulates in the lower part of the mask without succeeding in passing through the three layers, the projection resistance property is preserved according to the standard (Figure 19). After more washing cycles, the accumulation of blood within the inner layers is such that it can pass through the internal barrier in case of pressure or buffering. Results of the test carried out according to ISO 22609 are then negative.

To explain this property loss, the contact angle between a drop of synthetic blood and a mask was measured on new and washed IIR masks (Reference n°30). It appears that washing cycles change the surface properties of the outer layer of masks and that the contact angle θ rapidly evolves from values greater than 90° (hydrophobic behavior) to angles lower than 90° (hydrophilic behavior) for a washed mask (Figure 20). These modifications of surface properties, in agreement with previous conclusions on the fiber state of charge, lead to a loss of the projection resistance ("R" function) after few washing steps, whatever the type II-R mask brand used. The projection resistance cannot be claimed after a washing treatment and wearing a washed IIR masks in an operating room should therefore be proscribed for medical staff.

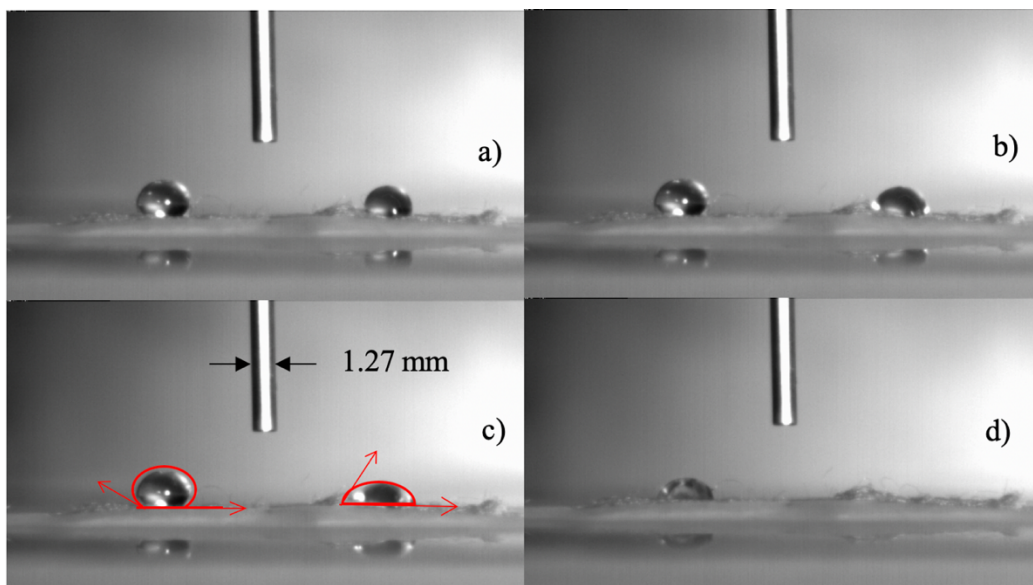


Figure 20: Comparison of contact angles of blood drops and mask surfaces for new (left) and 9-time washed masks (right) after a) 2 s; b) 30 s; c) 2 min 30 s and d) 5 min

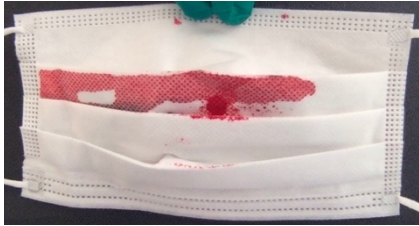








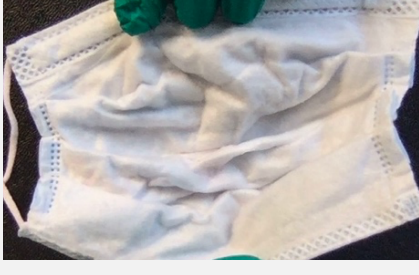


Ref.	Washing cycle	Outer face	Inner face
22	0		
22	2		
22	4		
32	1		
32	4		
32	9		

Figure 19: Examples of outer and inner faces of masks after a synthetic blood projection

Conclusion

Comparison of a large number of masks highlighted a large variability of PFE and breathability depending on the mask trademark, both for surgical masks (regardless of type) and for FFP2s. The quality of the meltblown and spunbond layers and the absence of electrostatic charges at the fiber surface can explain the lower fractional efficiency of some references. For medical face masks, even if the performances seem to be independent from the mask type, results tend to highlight that types II and IIR exhibit a similar behavior, both for the breathability and for the efficiency for 3 μm particle diameter. Washing, probably the most easily adaptable treatment for the general public, was the solution adopted for the mask decontamination. It should be noted that, whatever the mask reference and the washing procedure, the first cycle induces a slight decrease of the differential pressure and of the collection efficiency. The performances of the washed masks were maintained up to 10 washing cycles and met the requirements of the standards. Nevertheless, a statistical Mann-Whitney U test showed that, for both the PFE and the breathability, the results obtained on new and washed masks cannot be considered significantly different for the majority of the trademarks. Moreover, if the PFE for 3 μm particle diameter remains greater than 95 or 98%, whatever the type of surgical mask and the number of washings, the performance of the masks is impacted by the washing for submicronic particles. This treatment leads to a loss of electrostatic charges during the washing cycle as confirmed by the results obtained on a mask discharged by immersion in isopropanol and measurements of fiber state of charge. The modifications of surface properties after a washing cycle also leads to a loss of the hydrophobic behavior of type IIR surgical masks which can therefore no more be considered as resistant to blood projections.

Washing surgical or FFP2 masks can be a convenient solution in case of shortage of these single-use devices, but also to reduce the consumption of plastic materials. As long as the head straps and the nosebands will not break, the protection level and the breathability of these masks remain similar to the performances of new masks. Nevertheless, the projection resistance cannot be claimed after a washing treatment and wearing a washed IIR masks in an operating room should therefore be proscribed for medical staff.

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Appendix 1: Surgical and FFP2 mask references

Trademark	Type	Ref.	Number of washing cycles						Washing protocol	
			0	1	2	3	5	10		
BYD CARE (YY/T 0969-2013)	I	01	X							
Moen (602A-01))	I	02	X							
Sunrise Nursing	I	03	X	X	X				W2	
LiangYa (DGTMY)	I	04	X	X					W2	
FITEXIN	I	05	X	X				X	W4	
MEDWELL	II	06	X							
CA Diffusion 1931	II	07-1	X	X	X			X	X	W2
CA Diffusion 1931	II	07-2	X	X				X		W4
ALLMED	II	08	X							
Henan YADU Industrial Co.	II	09	X							
SAVOY International	II	10	X	X				X		W4
WK Well Klean	II	11	X							
LyncMed (302089-CMA010)	II	12	X							
LyncMed (302089-CMA006)	II	13-1	X	X	X			X	X	W2
LyncMed (302089-CMA006)	II	13-2	X							
Naguma (NA-05)	II	14	X							
Saudel (85002)	II	15	X							
TD Professional 45455	II	16	X	X						W2
TSC	II	17-1	X	X						W3
TSC	II	17-2	X						X	W4
Global	II	18	X						X	W4
Kolmi OP'R (M36101-30)	IIR	19	X	X				X		W4
Kolmi OPAIR (M31101-30))	IIR	20	X	X				X		W4
Kolmi OpairONE (M34101-30)	IIR	21	X	X				X		W4
CA Diffusion 1960	IIR	22-1	X	X				X		W4
CA Diffusion 1960	IIR	22-2	X	X	X			X		W2
CA Diffusion 1960	IIR	22-3	X	X	X	X		X	X	W1
Ansell (Sandel)	IIR	23	X	X						W4
FCHA Fengchenhan	IIR	24	X							
Segetex-eif (M193-25)	IIR	25	X	X				X		W4
France Cardio (France)	IIR	26	X						X	W4
MIF Medical (WA-FM)	IIR	27	X							
Yongli (YLEN104)	IIR	28	X							
Xiantao Xingrong (XR001)	IIR	29	X							
Jiangxi Hongda (Hygial)	IIR	30	X							
LCH (Aerokyn PLM.01R)	IIR	31-1	X	X	X			X	X	W2
LCH (Aerokyn PLM.01R)	IIR	31-2	X	X	X					W2
Medicom (2015-30)	IIR	32	X	X	X			X	X	W2
Paul Boyé (MPB-CH1)	IIR	33	X	X	X					W2
Kimberly Clark (The Lite One)	IIR	34	X							
Solida	IIR	35	X	X						W3
Ultrafilter (Ultramask EASM 198R)	IIR	36	X							

Trademark	Type	Ref.	Number of washing cycles						Washing protocol
			0	1	2	3	5	10	
VALMY (VR202F)	FFP2	A	X	X					W5
KOLMI (OpAir Pro white)	FFP2	B	X	X					W1
KOLMI (OpAir Pro violet)	FFP2	C	X	X					W1
Paul Boyé (MPB2.1-B.27069-TU-00)	FFP2	D	X	X					W1