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Effect of dry heat treatment between room temperature and 160 °C on surgical mask

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Abstract

The Covid-19 crisis has led to a high demand and use of surgical masks worldwide, causing risks of shortages and pollution. Therefore, decontamination of surgical masks could be an opportunity to reduce these risks.

In our study, we applied dry heat to the masks for 15 minutes at different temperatures and studied the consequences of heat on surface chemistry and fiber morphology. We focus here on the effects of dry heat treatment on the masks and not on the verification of mask disinfection, which has been thoroughly studied in existing literature.

The masks that were heated to 70°C, 100°C, 130°C, 140°C, 150°C did not show significant changes at the nanometric scale and the standard deviation of the surface temperature of the worn masks is similar to that of the unheated control mask. However we show a slight heating altered the hydrophobicity of the surface, and induced a significative modification of the wetting angle of water droplets. The mask heated to 157°C has a higher surface temperature standard deviation and fused fibers are observed by scanning electron microscopy. The mask heated to 160°C melted and then hardened as it cooled making it completely unusable.

Introduction

The significant impact of the Covid-19 outbreak paralyzed many countries in the world, forcing them to lockdown the population in order to stop the spread of the epidemic. The use of surgical masks helped to slow down the epidemic [1] [21]. In february 2020, single-use face mask production in China increased to 116 million per day [2].

Three major types of masks can be used : "Type I" masks, "Type II" and "Type IIR" masks. Type I masks filter 95% of the bacteria from an aerosol of average size 3 µm ; type II masks filter 98% of the bacteria from an aerosol of average size 3 µm ; type IIR filter the same as type II masks but are also splash resistant [3] [4]. A surgical mask can be either type. Type IIR masks are dedicated to the medical personnel.

Surgical masks are composed of non-woven polypropylene ; they have three layers and three folders. The first and the third layer are the same and the second one is a filter. The filter is made of meltblown [5] [6] [7] and is charged during the fabrication in order to increase the filtration capacity. Both the first and the third layer are made of 30 µm diameter fibers, the second one is made of 5 µm diameter fibers [8]. The daily use of the masks triggered a massive micro-plastic pollution [2]. According to the Oceanasia association, 1.56 billion face masks entered the oceans in 2020. Once in the oceans, these masks disintegrate into micro-particles which significantly disrupt the local ecosystem [9].

Thus, the reuse of surgical masks has been envisioned by many scientists. Various techniques have been tested: UV treatment [10] [11] [12] [13] [14], steam [15] [16] [14] [12] [10], dry heat [15] [14] [17] [10] [12], solvent [14] [12] [10]. According to the different studies, dry heat seems to be the easiest method to implement. It is known that the virus dies when

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exposed to a 63°C environment for 4 minutes [18]. However, decontamination techniques can lead to a degradation and a loss of efficiency of the masks [19]. The origin of the loss of filtration efficiency is not systematically explained. Indeed, this degradation may be due to a modification of the microstructure and the surface chemistry of the masks.

The objective of this study is to focus on the possible degradation of masks (and the causes of this degradation) which have undergone a dry heat treatment.

Methods

Two face masks EN 14683/2019 were heated in an oven at temperatures of 70°C, 100°C, 130°C, 140°C, 150°C, 157°C and 160 ° C, for 15 minutes. Wetting angle measurements, thermal camera temperature measurements, and Scanning Electron Microscope (SEM) observations were then performed to assess the impact of dry heat on the mask structure at the micro and nanometric scale. For each heating temperature, a mask was used to perform thermal camera measurements and the other was used to perform both wetting angle and SEM measurements. After being heated at 70°C, 100°C, 130°C, 140°C, 150°C, 157°C and 160°C, the wetting angle of the masks, the surface temperature and the microstructure of the masks were observed.

The wetting angle (β on Figure 1 *(a)*) measurements were carried out with a tensiometer. Small squares of masks were cut (around 1 cm^2) in each layer. The droplet had a constant volume of 1 µL except for the second layer where the droplet had to fall because of the high hydrophobia of the second layer. The measurements were repeated five times for each layer.

The InfraRed (IR) measurements were achieved with an iPi400 camera. A Region Of Interest (ROI) was chosen (the white square presented in Figure 2 *(a)*) and the mean surface temperature of the ROI was measured during 40 seconds. This experiment was repeated 5 times for each mask with the same ROI and the same subject. Then, data were extracted and processed to extract the standard deviation of the temperature.
The SEM images were taken with a TESCAN MIRA3 microscope. Small squares of masks were cut (around 0.25 cm^2) in each layer. The samples were placed on double-sided sticky carbon pads, which were sticked to an aluminum plane. The samples were then gold-coated by a sputtering device. The images were taken with an accelerating voltage of 10 kV.

Results

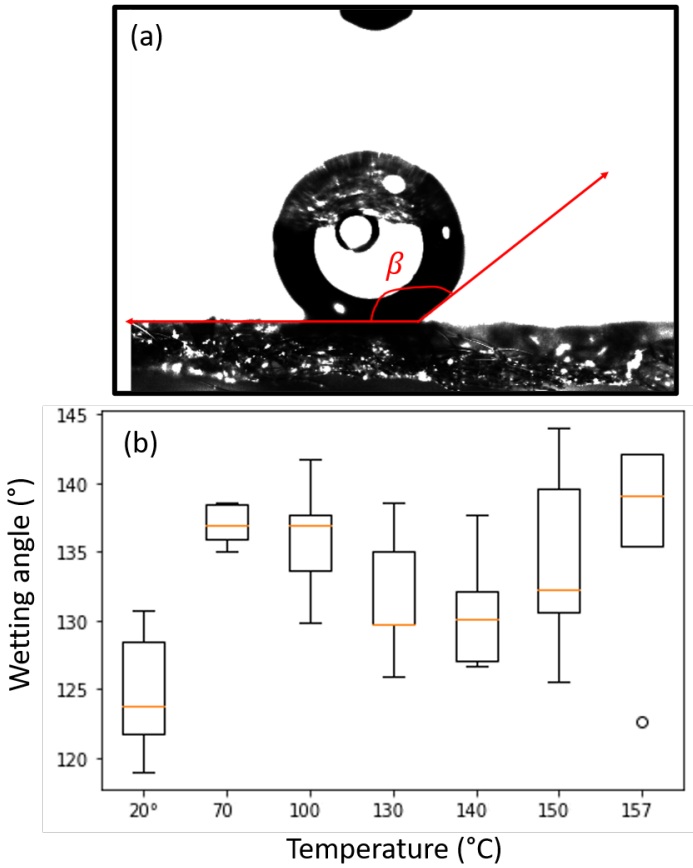

Figure 1: *(a) Measurement of the wetting angle with a tensiometer, (b) statistic distribution of the wetting angle for each mask*

Figure 1 *(a)* shows a typical liquid droplet wetting on the mask surface and the corresponding wetting angle β. The statistic distribution of the wetting angle is shown in Figure 1 *(b)*. The mean wetting angle increased going from 123°, with a standard deviation of 5° for the control sample to 137° and a standard deviation of 3° for the mask heated at 70°C. A significant increase of the wetting angle was observed for each heated mask. It should be noted the contact angle values were not significantly altered by series of annealing performed at the same temperature.

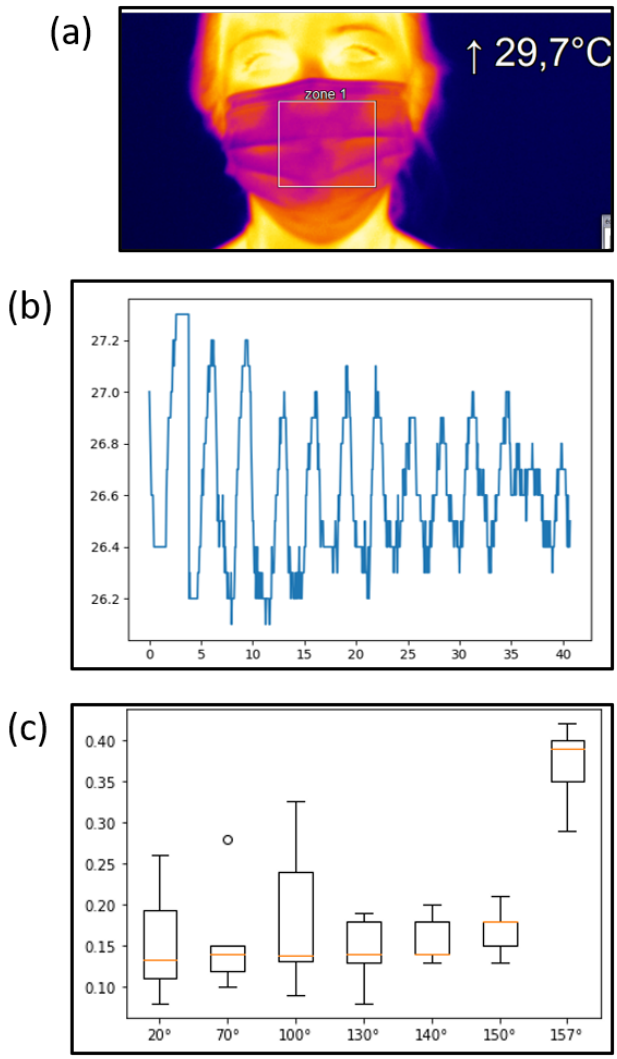

Figure 2: *(a) ROI observed with the IR camera, (b) Evolution of the surface temperature of the mask heated at 70°C as a function of time, (c) Statistic distribution of the Standard Deviation of the Surface Temperature (SDST) for each mask*

Figure 2 *(b)* represents the average temperature of the ROI of the mask heated at 70°C as a function of time. The maximum corresponds to the exhale of the subject and the minimum corresponds to the inhale of the subject. For the mask heated at 70°C, the mean temperature was 29°C and the standard deviation was 0.277 °C.
The statistic distribution of the standard deviation of the surface temperature (SDST) is represented in Figure 2 *(c)*. The SDST is stable for the temperatures until 157°, when it brutally increases. Indeed, for the control sample the SDST was 0.12°C and it was 0.39°C for the mask heated at 157°C.

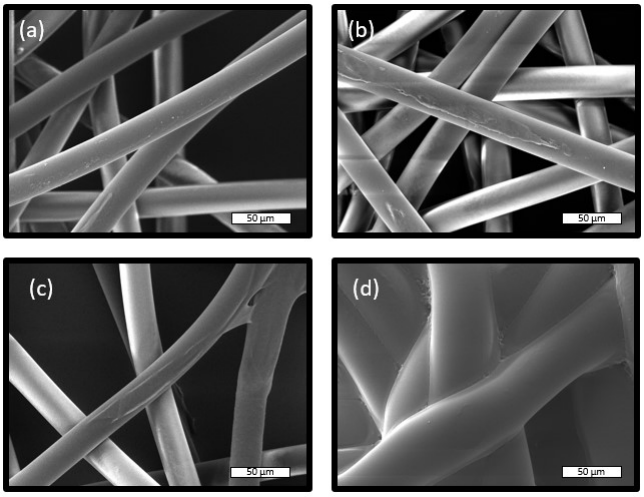

Figure 3: *(a) Control sample, (b) mask heated at 70°C, (c) mask heated at 155°C, (d) mask heated at 160 °C. (The magnification is 1.12k, all the images correspond to the first layer of the mask)*

Figure 3 *(a)* and *(b)* show the first layer of the mask of the control sample and of the mask heated at 70°C. Up to 155°C no notable changes were observed, the fibers looked like the control sample. However, for the mask heated at 155°C (Figure 3 *(c)*) the fibers partially fused. For the mask heated at 160°C, the temperature was so close to the fusion temperature that the fibers totally fused. All of the layers fused together, making the mask very hard and unwearable.

The following table synthesizes the results (a = means no notable changes compared to the control sample, a + means an increase and a × means that the measures could not be performed).

Mask	SDST (°C)	Wetting angle (°)	Visual aspect	SEM (fibers)
control sample	0.12	130	Normal	Intact
70°C	=	+	=	Intact
100°C	=	+	=	Intact
130°C	=	+	=	Intact
140°C	=	+	=	Intact
150°C	=	+	=	Intact
157°C	+	+	Blistered	Partly Fused
160°C	×	×	Fused	Totally fused

Table 1: *Table of results*

Discussion

The masks heated at the highest temperatures were deeply damaged by the treatment. Indeed, the fibers fused. Regarding the mask heated at 157°C, the fibers partially fused (Figure 3 *(c)*), making breathing harder for the user (which could explain the higher air flow coming outside from the mask). The mask heated at 160°C was unusable, as it had totally fused it was impossible to wear it. The increase in the airflow coming outside of the mask is not the only notable change.

The wetting angle increased significantly on the heated masks (even the one with the lowest temperature). This result can explain the source of the loss of filtration efficiency. Indeed, an increase in wetting angle means that the treated masks are more hydrophobic, which is important to prevent contaminated drops from reaching the user. However, this increase may be due to the loss of surface charges [20], that is likely to modify the surface's chemical properties. Surface charges are useful, as they increase the filtration efficiency of the masks. Thus, the filtration efficiency might have decreased during the decontamination process. As the filtration efficiency was not measured, this loss cannot be quantified.

To conclude, when heated, the masks might loose a portion of their surface charges, making them more hydrophobic but less efficient. The only notable changes in airflow coming outside of the mask were visible on the masks heated at 157°C and 160°C. This variation is due to the partial (or total) fuse of the fibers. The impact of a dry heat on face mask seems to be limited to a slight modification of its hydrophobic properties. However other characterization should be performed to ensure the mask integrity.

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