Universidade do Minho
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RESUMO

Estudo das propriedades do PDMS e a sua caracterização para o fabrico de máscaras faciais transparentes

A doença coronavírus (COVID-19) é provocada pelo vírus SARS-CoV-2 e foi declarada como pandemia pela Organização Mundial da Saúde (WHO) em março de 2020. De forma a prevenir a transmissão do vírus, a WHO publicou algumas precauções que toda a comunidade deverá considerar, tais como a utilização de máscaras faciais. Quando usadas por longos períodos de tempo, podem ser bastante desconfortáveis, deixando marcas na cara e até mesmo provocando hematomas. Os profissionais de saúde são mais vulneráveis a este problema. Adicionalmente, a utilização de máscaras afeta a comunidade surda, uma vez que limita profundamente a sua capacidade de comunicação. Tendo em conta estes problemas, surgiu a ideia de desenvolver uma máscara facial segura, confortável e transparente. Assim, o objetivo desta dissertação visa o desenvolvimento de uma máscara inovadora e diferenciadora utilizando um material tecnologicamente avançado, o polidimetilsiloxano (PDMS), que contém um conjunto de propriedades únicas comparativamente aos materiais tradicionais. Desta forma, foram desenvolvidas máscaras reutilizáveis a partir da combinação de tecido com uma janela transparente de PDMS. Foi, ainda, desenvolvido um método para reciclar PDMS, de forma a reutilizar este material no fim de vida da máscara, diminuindo assim a produção de resíduos. As propriedades mecânicas e óticas do PDMS nativo e reciclado foram estudadas para avaliar a sua adequabilidade para integração em máscaras. Os estudos realizados permitiram confirmar que o PDMS é um excelente candidato para este tipo de aplicações.

Adicionalmente, as máscaras produzidas foram enviadas para um laboratório de certificação, que classificou a máscara de PDMS nativo como nível 2 para uso geral, e a máscara de PDMS reciclável como nível 3 para uso geral. Assim, nesta dissertação é apresentado um processo de fabrico simples e económico para a produção de máscaras transparentes. As máscaras resultantes são adequadas para produção em massa e poderão ser lançadas no mercado a um custo economicamente viável.

PALAVRAS-CHAVE: CERTIFICAÇÃO, COVID-19, MÁSCARA TRANSPARENTE, PDMS, PDMS RECICLADO.

ABSTRACT

Study of the PDMS properties and their characterization for the fabrication of transparent face masks

Coronavirus disease 2019 (COVID-19) is caused by SARS-CoV-2 virus and it was declared as a pandemic by World Health Organization (WHO), on March 2020. In order to prevent the virus transmission, WHO published some simple precautions for the public to take in consideration, such as wearing facial masks. When used for long periods of time, masks can be very uncomfortable, leaving marks on the face and even causing wounds. The people who are more vulnerable to this problem are healthcare professionals. Additionally, the use of conventional masks also affects deaf community, as it profoundly limits their ability to communicate. Based on these problems, the idea of developing a safe, comfortable and transparent mask emerged. Hence, the objective of this dissertation is to develop an innovative and distinct mask using a technologically advanced material, the polydimethylsiloxane (PDMS), which has outstanding properties in comparison to traditional materials. Thus, reusable masks with fabric and a transparent PDMS window were developed. Besides, a method to recycle PDMS was developed in order to reuse this material at the end of mask's life, thus reducing the waste production. Optical and mechanical properties of native and recycled PDMS were studied to assess its suitability for integration in masks. The studies confirmed that PDMS is an excellent candidate for this type of application.

Additionally, the developed masks were sent to a certification laboratory, which certified the native PDMS mask as a level 2 for general use and the recyclable PDMS mask as a level 3 for general use. Thus, this dissertation presents a simple and cost-effective manufacture process to produce a transparent mask. The resulting masks are suited for mass production and can be launched in the market for an economically viable price.

KEYWORDS: CERTIFICATION, COVID-19, PDMS, RECYCLED PDMS, TRANSPARENT MASK.

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List of Acronyms

BFE - Bacterial Filtration Efficiency

DGS - Directorate-General for Health

EO - Ethylene Oxide

FFR - Filtering Facepiece Respirator

HPP - Hydrogen Peroxide Plasma

NP - Nanoparticle

PDMS - Polydimethylsiloxane

ROS - Reactive Oxygen Species

UV - Ultraviolet

VHP - Vaporized Hydrogen Peroxide

WCA - Water Angle Contact

WHO - World Health Organization

1. INTRODUCTION

In the present section, a contextualization on preventive measures for Coronavirus disease 2019 (COVID-19) is given, highlighting the need of developing transparent and comfortable face masks. Next, the motivation and proposed objectives for the work performed on the present dissertation are described. Finally, the dissertation's structure is presented.

1.1 Contextualization

COVID-19 is caused by SARS-CoV-2 virus and it is transmitted mainly through respiratory droplets [1]. On March 2020, World Health Organization (WHO) declared this disease as a pandemic and to prevent its transmission, this organization established preventive measures, such as physical distancing, wearing a face mask, keeping rooms ventilated, avoiding crowds, cleaning hands frequently, and coughing into a flexed elbow or tissue [1,2]. The use of masks have demonstrated to have a crucial role in reducing transmission and spreading rate of the virus [2]. However, using face masks has its drawbacks, mainly on a social and communicative level [3]. Having half of the face covered is a communication obstacle, in particular for deaf people, who depend on lip-reading for communication [4]. Additionally, when used for long periods of time, face masks can be very uncomfortable, leaving marks on the face and even causing wounds [5]. To overcome these limitations, several authors have reported the development of transparent masks. In fact, there are already transparent masks on the market [6,7]. However, almost none of those masks are classified as medical device, which means that healthcare professionals cannot wear them. It is crucial to develop a mask that is both transparent and comfortable and, additionally, that can be classified as medical device, providing a higher level of protection. Furthermore, the conventional masks are not recyclable and end as a waste on rivers and oceans. So, in order to reduce the material waste, as well as the environmental impact, and cost of masks, the fabrication of recyclable masks also represents a huge advantage.

1.2 Motivation and objectives

As exposed in the previous section, there are some limitations that need to be overcome in order to produce a transparent face mask that is suitable to the entire community during their daily lives. So, this project aims to contribute for the development of a transparent, reusable, respirable and biocompatible mask made with polydimethylsiloxane (PDMS) in combination with fabric, that fulfils all the requirements for a safe use, both in social and healthcare facilities context.

PDMS is an elastomeric polymer with interesting properties for biomedical applications, including: excellent resistance to biodegradation, biocompatibility, chemical stability, gas permeability, good mechanical properties, excellent optical transparency and simple fabrication by replica moulding [8–12]. One of the mechanical properties of PDMS is its flexibility [13], which allows a conformal contact with the face, providing a good isolation from the surrounding air and a greater level of comfort. Moreover, several methods can be used to sterilize PDMS, such as ultraviolet (UV) sterilization, allowing its reuse. Additionally, PDMS properties can be tailored in order to achieve other characteristics [14]. For example, when PDMS is mixed with different amounts of curing agent, its mechanical properties change. With such outstanding properties, PDMS seems the ideal material to produce a transparent mask. Combining the possibility of recycling associated with its transparency, flexibility, reuse, and breathability, it will consist in the first mask, on a worldwide level, with all these characteristics together. The main goals for the present dissertation are:

- Research existing transparent masks, their advantages, and limitations;
- Research technologies to improve PDMS features;
- PDMS characterization in order to verify its viability to be used as main material for a mask;
- Project and fabrication of a mask made with PDMS and fabric;
- Investigate a method for recycling PDMS.

1.3 Dissertation Outline

This dissertation is organized as follows:

- Chapter 1 presents the introduction and motivation for the selected subject, and corresponding objectives. Lastly, the dissertation structure is stated;
- Chapter 2 describes the state-of-art, where technologies for application in a transparent mask are described. More specifically, in this chapter the commercially available transparent masks are presented, as well as their advantages and limitations, PDMS properties, superficial modification for PDMS, sterilization methods and antibacterial treatments;
- Chapter 3 presents all the methodology adopted to project and fabricate the mask. PDMS fabrication process is enlightened as well as the used techniques to characterize its properties. Moreover, the procedures adopted to develop a face mask with PDMS, native and recycled, and fabric are explained;
- Chapter 4 shows the results and discussion of the performed tests regarding native and recycled PDMS and fabric. Additionally, the certification obtained for the developed mask is demonstrated;
- Lastly, in chapter 5, the conclusions regarding the work carried out, as well as the future work that will follow this dissertation are presented.

2. STATE-OF-ART: TECHNOLOGIES FOR APPLICATION IN A TRANSPARENT MASK

In this chapter, topics that are key for understanding the state of development of transparent masks and the limitations that must be overcome are presented. First, it is introduced the set of requirements to fabricate a face mask as well as the existing transparent masks on the market or under investigation. The following sections highlight the excellent features of PDMS and of recycled PDMS, showing their potential to be applied in the fabrication of transparent masks. Additionally, some anti-fog treatments for PDMS are presented as well as several sterilization methods. The last section of the state-of-art shows some antibacterial treatments for PDMS.

2.1 Masks and half-masks requirements

Given the need to develop new face masks that reach a vast world population for routine use, it is necessary that they provide a higher efficiency of protection, comfort, and transparency as well as the possibility of being reused and recycled. To begin, it is important to establish the difference between a filtering facepiece respirator (FFR) and medical mask. A FFR, or a respirator, or half-mask must be made with a filtering material or possess an exhalation valve made with filtering material [15]. KN95 is a commonly used FFR. A medical mask is made with non-woven fabrics and it is typically known as surgical masks. In this regard, to ensure that face masks and respirators are safe, strict testing and requirements have been set in place. EN 149:2001+A1:2009 [16] is a European standard that should be followed for fabrication of respiratory protective devices and filtering half-masks to protect against particles. This standard brings together the tests, requirements and marks that should be applied to the respirator in order to ensure its safe use. Respirators are classified based on their performance according to various assays namely, visual inspection, conditioning, practical performance, leakage, carbon dioxide content of the inhalation air, strength of attachment of exhalation valve housing, clogging, penetration of filter material, breathing resistance and flammability.

A FFR, or respirator, covers the nose, mouth, and chin, and can have an expiration valve. Additionally, a proper seal should be ensured between the respirator and the face. The EN 149:2001+A1:2009 [16] standard classifies FFR's as FFP1, FFP2 and FFP3, based on their

filtration efficiency and their total inward leakage (Table 1). A commonly used FFR is KN95 mask which are classified as FFP2.

Table 1. *Classification of filtering Respiratory Protective Devices (RPD) (adapted from [16]).*

Class	Efficiency	Total inward leakage (tested in laboratory)	Penetration of filter material (maximum %)
FFP1	Low	22 %	20 %
FFP2	Medium	8 %	6 %
FFP3	High	2 %	1 %

In addition to the FFR's, there are also medical masks (commonly known as surgical masks), which are certified by EN 14683:2019 [17] standard, that distinguishes three types of masks (type I, type II and type IIR), based on their characteristics, as follows: bacterial filtration efficiency (BFE), differential pressure (air permeability by the mask), microbial cleanliness (bioburden) and splash resistance. Table 2 summarizes the performance characteristics for each type of mask.

Table 2. *Performance characteristics for each type of mask, based on the EN 14683:2019 standard (adapted from [17]).*

Test	Type I	Type II	Type IIR
Bacterial filtration (BFE), (%)	$\geq$ 95	$\geq$ 98	$\geq$ 98
Differential pressure (Pa/cm^2)	< 40	< 40	< 60
Splash resistance pressure (kPa)	Not required	Not required	$\geq$ 16,0
Microbial cleanliness (cfu/g)	$\leq$ 30	$\leq$ 30	$\leq$ 30

Table 3 summarizes a categorization system, suggested by Directorate-Generate for Health (DGS) together with food and economic safety authority (ASAE), Portuguese Institute of Quality (IPQ) and National Authority of Medicines and Health Products, I.P. (INFARMED), that refers which type of mask should be worn, taking in consideration the goal:

Table 3. *Mask categorization system based on their goal* [18].

Type of user	Type of mask	Regulatory qualification
Healthcare professionals and patients	Half-masks for respiratory protection (FFP2, FFP3).	PPE
	Type I and type IIR medical masks. Non reusable.	Medical device
Professionals who are in frequent contact with public	Type I medical masks. Non reusable.	Medical device
	Alternative masks for frequent contact with public, non-reusable or reusable.	Textile product
Professionals who are not teleworking or general population	Alternative masks for infrequent contacts, non-reusable, or reusable.	Textile product

Despite the existence of several masks that meet the previous mentioned standards, they still present some limitations, such as: the need to be frequently replaced, since there are not sterilization methods that maintain the characteristics of the mask without degrading it; the use for long periods of time showed to have led to discomfort by the user and even to cause wounds on the skin; they are not recyclable which increases the environmental impact and, above all, they are, usually, opaque which is unsuitable for interpersonal recognition.

Additionally, the transparency is an essential feature for the deaf community, since wearing a non-transparent mask deeply limits their communication capability.

2.2 Transparent masks

As exposed in the previous section, people who heavily depend on lip-reading for communication have been adversely affected by the pandemics [4]. For that reason, transparent masks have been developed so that this adversity is overcome. A transparent mask needs to be nonpermeable to liquids and fluids and to be breathable for human wear [19]. Those requirements need to be fulfilled whilst the protection against the virus is guaranteed. Herein, a review of the transparent masks will be made.

The search for transparent masks did not start with COVID-19 pandemics. For a long time, researchers have shown interest in developing innovative transparent face masks, that fulfils the needs of the whole society. For example, in 2008, Hahne [20] developed a face mask with a central transparent portion, in order to promote better communication between healthcare professionals and patients, in hospital context. Similarly, Reese et al. [21] claims to have produced a medical facial mask with transparent portion that is suited to be used in medical context. More recently, commercially available product ClearMask^{TM} [22] (Figure 1) claims to be the first fully transparent face mask. Unlike the masks referenced above, this one does not have only a transparent window, it is fully transparent. A drawback of this mask is that the breathability is guaranteed through the sides of the mask, which means that the air is not properly filtrated. This mask costs 5.83 euros, which for a single-use mask is relatively over-priced. All the above-mentioned authors claimed that their mask have anti-fog properties and are suited to be worn by medical care workers. A drawback is that none of these masks are reusable.

Figure 1. *ClearMask* [6].

Another approach of transparent mask was developed by Rahmayanti et al. [23] who chose nata de coco to produce it. Nata de coco is the result from Acetobacter xylinum fermentation and, by controlling the pores between the Nata de coco fibbers, transparent masks can be obtained. This approach showed good permeability and transparency that can be applied in face masks. Although this strategy shows a great potential for the fabrication of transparent and breathable masks, the fabrication process is complex. The authors did not present any study about the efficiency of the mask. There are no reports on whether this mask has efficient filtration or is breathable. In a similar way to the previous masks, this one is not reusable.

Healthcare workers must wear FFR's instead of surgical or cloth masks [15]. An example of these respirators is N95 masks, which offer more protection against pathogens compared to the normal disposable surgical masks. Respirators usually offer a better fitting and have higher filtration capability when comparing to surgical masks [15]. Therefore, in addition to the transparent masks mentioned above, transparent elastomeric respirators have also been developed. For example, Wentworth et al. [24] have developed a transparent, elastomeric, adaptable, long-lasting (TEAL) respirator. Figure 2 shows a prototype of TEAL mask. As can be seen, breathability is guaranteed through the two filter pieces. The authors mentioned that

their mask has not yet anti-fog properties, but they are working on it. Elastomeric respirators have the advantage of being reusable since they are cleanable and/or sterilizable.

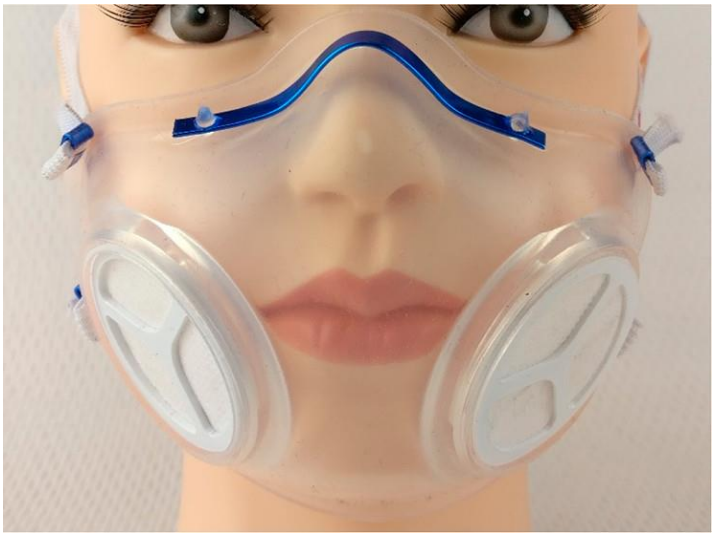


Figure 2. *Prototype of TEAL mask* [24].

Alenezi et al. [19] proposed a reusable face mask with square-waveform design. They used a clear epoxy resin as main material to achieve a good transparency. The mask contains two filter caps with filter material that can be removed and cleaned or replaced. The authors showed the effectiveness of the mask through numerical simulations, where they proved that the mask has the ability to filter airborne pathogens and to breath without difficulty. Figure 3 represents the product development procedure.

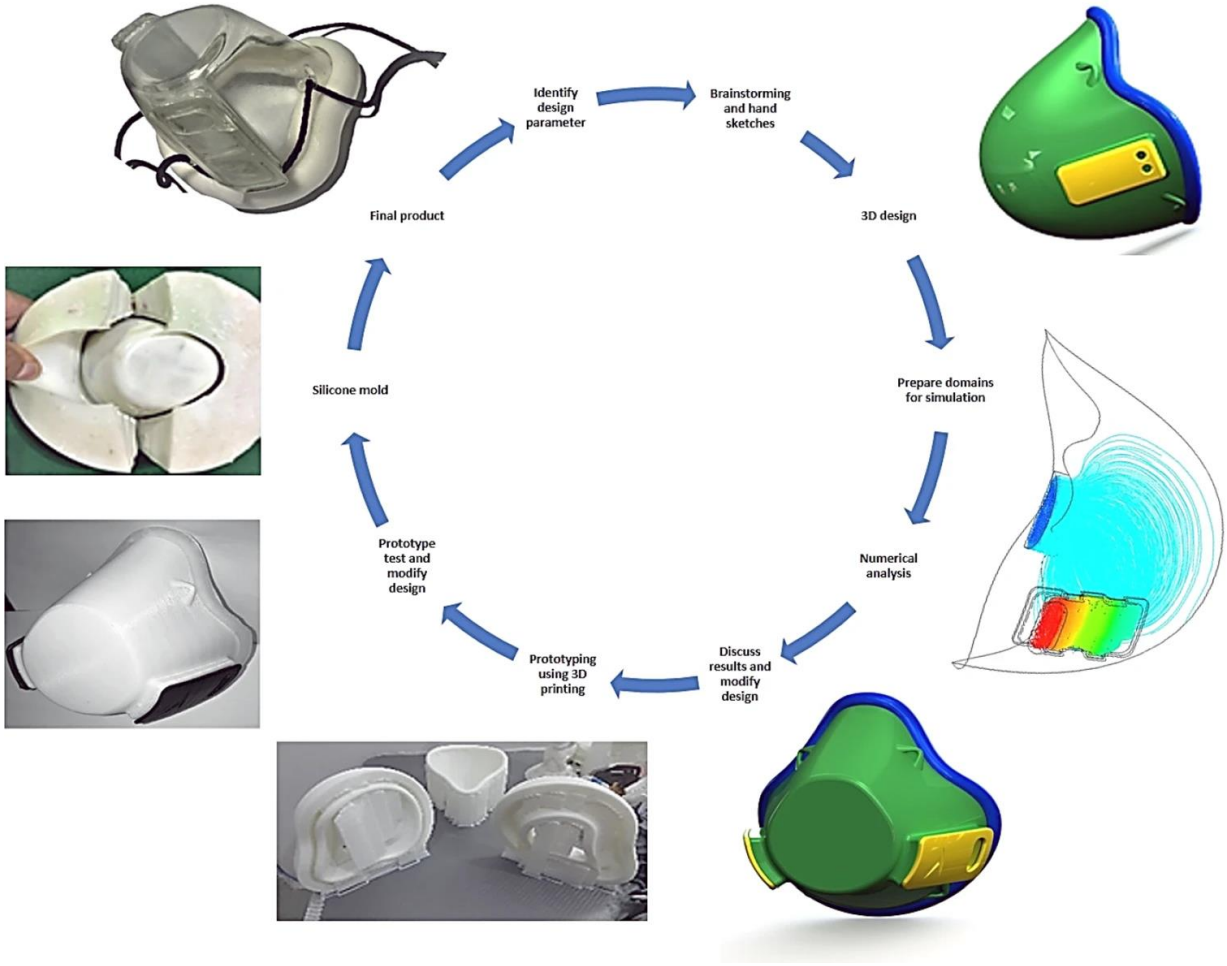


***Figure 3**. Process flowchart for the initial design and product development procedure using CAD, flow simulation, 3D printing, and cold moulding processes* [19].

Recently, Redcliffe Medical Devices inc [7] claimed to have developed the world's first and most effective, self UV-C sterilizing mask with N100 HEPA filtration. The company have now commercially available three types of respirators: Leaf HEPA, Leaf UV (Figure 4) and Leaf PRO. All varieties of the Leaf Masks are made with an optical grade silicone and have a modular exhaust system. The exhaust system can be upgraded with N95 filters to provide a better filtration. Leaf UV mask possess a UV-C sterilization system that begins ten minutes after the mask is taken off and plugged into a USB power source. The sterilization takes two minutes. This mask certainly presents an outstanding design. Despite its great features, the main drawback of the masks is their over-price (for example, Leaf UV mask costs 99 american dollars), limiting its widespread use.

Figure 4. *Leaf UV mask* [7].

Despite the advances that have been made to develop transparent mask that can be worn by both medical personnel and citizens, it is still necessary to continue to develop new advanced masks. There is not yet a mask that can fulfil all the following requirements:

- Transparent;
- Reusable;
- Low-cost;
- Easy fabrication;
- Possibility of mass production;
- Classified as medical device;
- Anti-fog properties;
- Recyclable.

Therefore, it is essential to develop an innovative mask capable of overcoming some of these limitations through the investigation of new materials and fabrication methods as well as anti-fog solutions so that the requirements above can be fulfilled. Additionally, PDMS have shown to be a technological advanced material with outstanding properties for the development of transparent masks.

2.3 PDMS properties

PDMS have unique and advantageous properties such as transparency, biocompatibility, gas permeability, flexibility allowing conformal and comfortable contact with skin face, supports sterilization processes allowing its reuse, and it has a fast, simple and low-cost manufacturing process [8–12]. In addition, PDMS presents a variable elasticity; its Elastic Modulus is 1−3 MPa [25–27], can work as a thermal and electrical insulation and degrades quickly in the natural environment [28]. PDMS presents a hyperelastic behaviour, which is the ability of a material to undergo large deformations before rupture [29]. This characteristic is also found in biological tissues and, for that reason, PDMS is a well-suited material to mimic, for example, blood vessels [28]. Another characteristic of this elastomer is its biocompatibility, which means that PDMS is compatible with biologic tissues [28]. PDMS presents a transmittance up to 90% at the spectral range from 390 nm to 780 nm [30–32] and, due to these characteristics, PDMS is a well-suited material to be integrate into transparent masks. In Table 4 some physical properties of PDMS are listed.

These characteristics offer the possibility for the integration of electronic circuits in a PDMS mask, such as a control UV-C sterilization system, a cooling system, and sensors to measure vital signals (O_2, temperature, heart rate, blood pressure, among others). Furthermore, the PDMS surface could be chemically/physically modified to ensure an anti-fog coating. Moreover, due to these characteristics, PDMS has been widely used in micropumps [33], catheter surfaces [34], dressings and bandages [35], microvalves [36], optical systems [37,38], in the *in vitro* study of diseases [39,40], in implants [41,42], in microfluidic and photonics [26,43–45].

Based on the characteristics listed in Table 4, PDMS shows great potential to be used as the main material for a transparent mask fabrication. Additionally, it is important to notice that the use of face masks have been a crucial factor on the environmental pollution, as reported by many authors [56–59]. This is an aspect that must be taken in consideration when projecting a face mask. Stevens et al. [60] presented a study about the impact of PDMS in the marine environment and concluded that PDMS is not a pollution factor. However, it would still be relevant to decrease the waste at the end of product's life through recycling. According to Table 4, PDMS melting point is around -45 °C, which means that melting it for reuse would

not be a feasible solution. A possible solution would be to reduce it to powder so that it could be mixed with new PDMS before curing.

Table 4. *Typical properties of cured PDMS.*

Property (unity)	Result	References
Transmittance at range 390 nm to 780 nm (%)	75–92	[46,47]
Index of refraction	1.4	[48]
Thermal conductivity (W/m·K)	0.2–0.27	[49,50]
Specific heat (kJ/kg·K)	1.46	[48]
Dielectric strength (kV/mm)	19	[49]
Dielectric constant	2.3–2.8	[48]
Electrical conductivity (Ω·m)	4×10^{13}	[48]
Volume resistivity (Ω·cm)	2.9×10^{14}	[49]
Young's Modulus [kPa]	360–870	[51]
Poisson ratio	0.5	[52]
Tensile strength (MPa)	2.24–6.7	[48,49]
Hardness [Shore A]	41–43	[47,53]
Viscosity (Pa·s)	3.5	[49]
Hydrophobicity – contact angle (°)	~108±7	[54]
Melting Point (°C)	-49.9–40	[55]

Despite the advantages, PDMS has some properties that can present a limitation in some applications. Due to its CH_3 groups, PDMS presents a hydrophobic surface (contact angle with water ~108°±7°) [54,61,62], often limiting its application in solutions composed of biological samples [63]. Additionally, PDMS tends to swell when combined with certain reagents [27,44]. One of the problems encountered in the constant use of elastomeric masks is related to the fogging and condensation of water droplets expelled during breathing. Thus, a chemically/physically modification of the hydrophobic characteristics of PDMS may be necessary to ensure an anti-fog or fog-resistant coating. In this regard, much effort has been made to make the PDMS surface hydrophilic [26,64–68]. Several hydrophilic treatments for PDMS are described in the following sub-section.

2.4 Surface modification of PDMS

As previously mentioned, due to the hydrophobic nature of PDMS, breathing causes fog on PDMS surface. The water contact angle (WCA) is commonly used to describe surface wettability, however the contact angle hysteresis also plays an important role on the droplet mobility [69]. Contact angle hysteresis is the difference between the advancing contact angle (θ_a) and the receding contact angle (θ_r) [70]. The lower the contact angle hysteresis, the higher is the droplet mobility [71]. PDMS presents a contact angle hysteresis of 2° [72], which means that the principal factor of impedance of polar liquids to flow may be the hydrophobic nature of PDMS. Based on this, it is important to study superficial modifications techniques that allow increasing the PDMS hydrophilicity.

Anti-fog coatings solutions can be of two types: superhydrophobic or hydrophilic [73]. A superhydrophobic coating is usually soap-based and works by repelling and spreading the fog moisture as it hits the surface. On the other hand, a hydrophilic coating acts like a microscopic sponge by interacting and absorbing moisture into the coating. Superhydrophobic coatings tend to wash off over time because they are soap-based. Therefore, hydrophilic coatings tend to last longer than the superhydrophobic [73].

Strategies employed attempting to solve PDMS hydrophobicity include surface activation methods such as: oxygen plasma; UV/ozone treatments; and corona discharges, which are widely used for PDMS surface oxidation to increase its wettability. The

main benefits of these methods are the short treatment time and easy operation, but the PDMS surface recovers its hydrophobicity when in contact with air within a few minutes [74–76].

Another method is physisorption, which is a simple and efficient approach that relies on surface hydrophobic or electrostatic interactions. This method includes the following techniques: layer-by-layer deposition; non-ionic surfactants; and charged polymers. The disadvantages are the lack of covalent bonds between PDMS and surface modifiers, which leads to the loss of modifiers quickly through desorption [77–79].

In order to improve the difficulties encountered in physisorption, chemical modification methods allow to maintain a long-term stability of the modified surface. These methods include: chemical vapor deposition; surface segregation and self-assembled monolayers; silanization; and polymer brushes via grafting methods [8,54,80–82].

Oxygen plasma is the most employed treatment to increase PDMS surface hydrophilicity because of its short treatment time, easy operation, and the fact that it does not affect the PDMS transparency [25,62,83,84]. However, this treatment is also known for losing its effects within minutes after exposure to air. For this reason, a variety of well-studied treatments have emerged with the purpose of overcoming this limitation [26,64–68]. Additionally, some articles reported that oxygen plasma may damage PDMS surface [54,85]. Therefore, Shin et al. reported three different treatments that do not require oxygen plasma pre-treatment, including Teflon coating, commercially available water-repellents and perfluorodecyltrichlorosilane (FDTS) [86]. The authors showed that the Teflon and the water-repellent decrease the hydrophobicity of PDMS with great chemical stability and without significantly affecting its transparency.

UV/ozone treatments and corona discharge are also commonly employed hydrophilic treatments, but as with oxygen plasma treatment, PDMS quickly recovers its hydrophobicity [54]. There have been efforts to improve some of these treatments however, the best way to achieve an effective and long-lasting treatment seems to be the combination of a surface activation with a covalent surface functionalization [26,85]. For example, Zhao et al. [87] proposed a method where PDMS is firstly activated by oxygen plasma treatment and then it is coated with a zwitterionic poly(methacrylate) copolymer (PMGT). This method allowed to decrease WCA of native PDMS from 108° to 30°, with a duration of at least

200 h. Equally, Zhou et al. [26] suggested a combination of gas-phase with wet chemical methods in order to achieve a better surface stability in a shorter treatment time. Examples of these treatments are combination of UV or plasma treatment and silanization, combination of UV or plasma treatment and graft polymerization, and combination of plasma treatment and layer-by-layer (LBL) assembly.

Although the methods listed above have been successful in improving the hydrophilicity of the PDMS surface, they have some limitations, such as chemical instability, need for specific equipment, limited manufacturing process for large scale, some methods cause loss of transparency, loss of mechanical properties and do not provide the hydrophilic surface for a long period of time [54,86]. Considering these facts, the work of Gökaltun et al. [88] presented a simplified method of easy manufacture, which uses copolymers composed of poly(ethylene glycol) and PDMS segments (PDMS- PEG) to reduce the hydrophobicity of PDMS without changing its transparency, biocompatibility and mechanical properties, with durability of 20 months. Other solution may be the use of an anti-fog or fog-resistant coating, such as a glycerine-based PDMS surface treatment, which prevents fogging on the inner surface of a PDMS mask [89]. Periodic pulverization should be employed to restore the hydrophilic surface.

On the other hand, if the goal is to increase PDMS hydrophobicity, adding waxes such as paraffin or beeswax to PDMS has been demonstrated to be capable of increasing the corrosion resistance, hydrophobicity, thermal and optical properties of PDMS, and using in superhydrophobic coating [90].

2.5 Sterilization methods

When developing a mask, it is important to consider its viability to be reusable. Consequently, it may be interesting to explore sterilization approaches that would allow recycling and reuse masks. Sterilization is, according to Cambridge Dictionary, “the process of making something completely clean and free from bacteria” [91]. This dissertation aims the development of a face mask fabricated with PDMS, therefore a research for sterilization methods that could be applied on PDMS was made. In the following sub-sections, some sterilization methods, their advantages, and limitations will be explained.

2.5.1 Sterilization methods with high temperatures

Sterilization methods with high temperatures can be of two types: dry heat and steam. Dry heat sterilization is an approach where the device or material is placed in an oven at high temperature (approximately 160 °C) [92]. Steam sterilization combines heat and moisture in an autoclave. This combination makes it easier for the heat to penetrate in the material, therefore, this technique usually requires lower temperatures and shorter times than the previous one (dry heat) [92].

These methods are suited for objects that can tolerate humidity and temperature (121 °C to 148 °C) [93]. Moreover, It was not found any evidence in the literature that this method affects PDMS properties since it is stable at temperatures up to 200 °C [49].

2.5.2 Ethylene oxide (EO) sterilization

EO is a colourless gas and the efficiency of the EO sterilization depends on the concentration of the gas, temperature, relative humidity, and gas exposure duration. The first step of this technique is to remove the oxygen from the sterilization chamber. In the second step, the sterilization chamber has to be heated (to temperatures in the range of 30 - 60 °C) and humidified. EO gas is injected, in the third step, at a pre-determined pressure. The first step is repeated in order to remove EO from the chamber. Finally, filtered air is bleed in the chamber to bring it to atmospheric pressure [92].

This technique is characterized by its effectiveness, and it is compatible with most materials. EO sterilization is the commonly chosen method for materials that are sensitive to heat, moisture, or radiation. Hence, this technique is suitable for thermolabile plastic and elastomer polymeric materials [94]. Additionally, it was found that this method is suitable for devices that contain electronic components. However, the vacuum may not be acceptable for embedded batteries [92–94].

One of the biggest limitations of this process is that toxic residues can remain in the sterilized material. However, this depends on the EO gas concentration, duration and absorptivity power of the polymers [92]. In Addition, EO sterilization is associated with lengthy cycles, high costs and potential hazards to patients, staff, and environment. EO gas is a flammable and explosive gas, hence it requires sophisticated equipment and trained personnel to handle it. It was found that overexposure to EO may result in irritation and

central nervous depression. For long-term exposures, it was found that EO can increase the risk of cancer [94].

2.5.3 Radiation sterilization

There are two types of radiation sterilization: gamma sterilization, which uses high energy gamma rays, and electron beam sterilization, that works with a constant steam of high energy electrons [92]. These approaches have the main limitation of using radiation since it is dangerous to humans. The standard dosage of these techniques is 25 kGy. However the lethal dosage of radiation to humans is about 0.01 kGy [95]. This means that radiation sterilization implies the implementation of shielding and robust interlocks in radiation processing. Additionally, when radiation comes into contact with oxygen, ozone is formed [95]. This requires the use of adequate ventilation and prevention of ozone inhalation. One of the biggest advantages of radiation sterilization is its simplicity and convenience for large-scale processing [95].

There are differences between these two types of radiation sterilization methods. For example, the distance between the device and the source of radiation is not a concern in the gamma method because gamma rays can travel long distances and still have the ability of penetrate the material. On the other hand, electron beam methods have penetration distance limitations, implying extra planning and care. Furthermore, electron beam sterilization is faster than gamma sterilization, although gamma rays have a greater capability of penetration than electron beam [95].

There is no evidence on the literature about if the radiation cause degradation on PDMS however, it is known that polymers such as glycolic acid (PGA), polymethyl methacrylate (PMMA) and polyvinylidene (PVF) are sensitive to radiation [92]. Gamma rays may affect polymers and semiconductors therefore, this method should not be applied to devices containing embedded electronic. The same is applied to electron beam sterilization, which causes damages on electronic components [93].

2.5.4 Hydrogen peroxide (H_2O_2) sterilization

There are two techniques associated with the H_2O_2: vaporized hydrogen peroxide (VHP) and hydrogen peroxide plasma (HPP). VHP sterilization is a method suitable to be applied on devices that contain electronic components. Additionally, it is known that the penetration of

VHP is lower than of EO gas. HPP consists of four stages: in the first stage, the sterilization chamber is vacuumed, the second stage consists of injecting H_2O_2, diffusion is performed in the third stage and finally, plasma is discharged. HPP sterilization is not appropriated for devices that contain electronic components because the plasma discharge stage produces high frequency energy [92,93]. However, VHP is well suited for devices containing embedded electronics, although vacuum can affect batteries lifespan [93].

H_2O_2 sterilization does not cause toxic residue accumulation which is an advantage over EO sterilization [92]. Additionally, H_2O_2 sterilization requires short exposure times, reducing the risk of toxic residues that could remain in the sterilized device [95].

H_2O_2 has a very high vapor and boiling point, requiring the use of deep vacuum pressures that may adversely affect materials [92,96]. In addition, this gas has a lower penetration capability than steam, dry heat or EO sterilization, being commonly characterized as a surface sterilant. Despite its final products be water vapor and oxygen, this sterilization approach begins with very hazardous highly concentrated H_2O_2 [96].

2.5.5 Ozone sterilization

Ozone sterilization is a method that starts with vacuum creation. After that, devices or materials are humidified, and ozone is created. Devices are exposed to two ozone cycles and then, ozone is removed from the chamber [92]. Usually, at the end of the process ozone is degraded in oxygen. Ozone is highly oxidative hence, the materials may be oxidative resistant [96]. This approach involves relatively low temperatures, making it suitable to be applied in heat sensitive materials [95,96]. Ozone sterilization produces no toxic residues and it has greater penetration capability than H_2O_2, but it is not as penetrable as EO, steam or dry heat [96]. No evidence was found in the literature about if PDMS reacts with ozone.

2.5.6 UV sterilization

UV rays can be used within a range between 200-280 nm to sterilize equipment. UV light has lower penetration power when compared to other techniques [92]. UV-C light has been widely used for disinfection against pathologies that are dangerous to humans, and it covers a range of the electromagnetic spectrum between 100 and 280 nm. The 222 nm wavelength has been mentioned as a germicidal wavelength that is not dangerous for humans, unlike the conventional 254 nm [97,98]. However, the majority of the commercial UV-C sterilizers use a

280 nm wavelength light. The efficiency of UV sterilization and the properties of the post-sterilization material depends on the time of exposure, on the wavelength used and on the intensity of the light [99]. Moreover, the adequate exposure time, intensity and wavelength of the light should be chosen accordingly to the material and to the microorganisms that is pretended to inactivate [99].

2.5.7 High intensity light or pulse light (PL) sterilization

PL sterilization is a technique that consists of emitting short duration pulses of intense, broad-spectrum light. UV light is commonly used in this method. This approach also has the limitation of having low penetration power however, it is suitable for surface sterilization [92]. PL sterilization strongly depends on the frequency and intensity of the PL and in certain circumstances, PL sterilization can be more effective in comparison with UV sterilization [100]. This method is a non-thermal method and produces no toxic residues [100].

2.5.8 Conclusions

There are two sterilization methods that seem to be suited for a PDMS mask: sterilization methods with high temperature and UV sterilization. DGS launched a manual with "General Measures for the Prevention and Control of COVID-19" [101], where they recommend washing clothes in a washing machine, at the highest possible temperature (at least 60 °C, for 30 minutes, or between 80-90 °C, for 10 minutes). Additionally, it was seen that PDMS is not affected by temperatures up to 200 °C [92]. UV sterilization also seems to be a well-suited method. There is commercially available equipment for UV sterilization that use a 280 nm wavelength light [99]. However, it is not known, yet, if this wavelength affects all the PDMS properties. Both solutions are suited to the daily use and available to all society.

2.6 Antibacterial treatments

It is expected that masks would be worn more than once and along the day. Because of this, it is important to consider that the mask will be exposed to a variety of microorganism that could be harmful to the human's health. Bacteria are an example of these harmful microorganisms, which can easily attach to solid substrates and create biofilms. Biofilms are more resistant than the bacteria alone therefore, it is important to create a mechanism capable of preventing the formation of biofilms [102].

Antibacterial coating act based on one of the following strategies: antibacterial agent release, contact-killing and anti-adhesion/bacteria-repelling. Antibacterial agent release-based coating act by leaching loaded antibacterial compounds over time. In contact-killing strategy, antimicrobial compounds are covalently anchored to the material surface, which induces cell membrane disruption. Anti-adhesion coatings imply physical surface modifications to prevent the bacteria to adhere on the surface [102].

Antibacterial coatings imply the use of antibiotic agents. Metal nanoparticles (NPs) have been studied because of their efficient antibacterial activity. Heavy metals are the common metals used on NP fabrication. However, they have the limitation of being toxic at high concentrations, but can serve the purpose of antibacterial activity in smaller amounts, without being toxic to humans [103]. In addition, a coating that is superhydrophobic is also going to help achieving a self-cleaning coating. In order to fabricate a superhydrophobic coating, two methods could be applied: optimizing roughness on a low surface energy material or modifying a rough surface using low surface energy materials. Superhydrophobic coatings can be achieved combining PDMS with TiO_2. There is a variety of studies that demonstrate the benefits and feasibility of the use of PDMS in superhydrophobic materials. However, some of the approaches imply the use of specific and expensive equipment or complicated procedures [104].

Antibacterial activity of NPs is mainly explained by their ability to generate ROS (Reactive Oxygen Species), which are destructible to bacterial cells. Ag metal is the most used metal for antibacterial treatment. However, many other metals have been used to fabricate NPs with the same purpose, including Al (Al_2O_3), Au, Bi, Ce, Cu (CuI, CuO, Cu_2O), Fe (Fe_2O_3), Mg (MgO), Ti (TiO_2) and Zn (ZnO) [103].

It is usual to add a capping agent during NP fabrication in order to increase stability and facilitate the dispersion of the NPs. Capping agent also contributes for the non-toxicity of the NPs, since it prevents NP agglomeration [103].

TiO_2 has been of a great interest as a filler in PDMS-based composites, due to its high permittivity, chemical inertness, non-toxicity and ease of dispersion. Therefore, it is important to understand the properties modifications that this combination could bring to the PDMS. For example, PDMS transparency must be maintained after any modifications. Vaimakis-Tsogkas et al. [105] studied the effects of the addition of TiO_2 on PDMS. They concluded that

this addition can, in fact, improve PDMS properties. The team observed that the addition of TiO_2 lowered the Young's Modulus and enhanced the strain of break of the PDMS. In addition, thermochemical properties, thermal stability and photostability were also improved. It was also shown that these coated films presented a high rate of NO oxidation, which is an important parameter, since the aim of the mask is to be used at the outdoor, and this property allows purification of the air.

Surface modifications combining TiO_2 thin films and hydrophobic agents have been studied, as they are a strategy capable of developing an efficient self-cleaning coating. Neves et al. [106] showed that TiO_2 thin films modified with PDMS can be used as a hydrophobic coating with photocatalytic activity. The team developed a film using vinyl-terminated PDMS without changes the optical properties of the final material. This film possesses photocatalysis and hydrophobicity properties, which are desired properties to achieve a self-cleaning surface.

Yousefi et al. [107] proposed a method where nanoparticles of TiO_2 are introduced in PDMS, making PDMS surface become superhydrophobic and with self-cleaning properties. The team observed that self-cleaning property is most probably due to the reduced chemical inhomogeneity at the surface layer of the superhydrophobic sample.

Wang et al. [104] also demonstrated that a PDMS solution blended with TiO_2 nanoparticles result in a superhydrophobic coating. ThIs coating has long-term durability against UV and sunlight and maintain their properties after long periods of outdoor exposure. Additionally, this coating is resistant to corrosion, high temperatures, and has high anti-impacting ability. The team could achieve a method with all these features and, in addition, the coating has self-cleaning properties.

2.7 Conclusions

The previous review intended to choose the best method to produce a transparent mask. Considering the transparent masks developed by the authors mentioned in section 2.2, it is clear that transparent respirators, like the ones developed by Wentworth et al. [24], Alenezi et al. [19] and Redcliffe Medical Devices inc [7], have a very time consuming and expensive manufacture process. Additionally, the manufacture processes mentioned are not ready for mass production. On the other hand, the masks proposed by Hahne [20] and Reese

et al. [21] have more simple manufacture processes and, therefore, they can be more cost-effective. Furthermore, if well planned and organized, those type of mask can be produced in mass. Based on this, a transparent and low-cost mask integrated with fabric that can be also reusable and recyclable will be projected in this dissertation. Although there are several masks in the market, none of them meets the characteristics of the mask developed in this dissertation.

The outstanding properties of PDMS combined with anti-fog and antibacterial solutions present a huge potential for the development of efficient face masks, that provide:

1. Contamination reduction;
2. Recognition between wearers and easy communication due to the PDMS transparency;
3. Capability of sterilization (withstand temperatures up to 200 °C) and reuse;
4. Ease of adjustment to any face, since PDMS is a light material, biocompatible and flexible;
5. Low-cost due to the simple and rapid fabrication process and the possibility of recycling and reuse.

This state-of-art will serve as guide to perform the tests described in the following sections. The information collected will allow to compare the results obtained in this dissertation with the ones in the literature. Moreover, this chapter allowed a better insight about the problematics associated with the existing transparent masks and will help in choosing the manufacturing processes.

3. METHODOLOGY

In this chapter, it is presented the PDMS fabrication process, as well as the characterization techniques for validation of PDMS as a suitable material for masks fabrication. All the procedures inherent to the development of PDMS-based mask, are described.

3.1 PDMS fabrication process

Sylgard® 184 Silicone Elastomer Kit is the most used commercial PDMS. It consists of a monomer and a curing agent, which are usually combined at a weight ratio of 10:1. The compound is mixed and then degassed with a desiccator, in order to prevent the formation of micro-bubbles. The PDMS solution is poured over the master mould and then cured in the oven [108]. The curing time depends on the temperature of the oven and on the size of the PDMS sample. The higher the hardening temperature, the less time it will take for the PDMS to cure. After the curing process, the piece is taken out of the mould [49]. Note that for very specific applications and complex geometries, it is usually advised to perform the curing process at room temperature for, at least, 48 hours [47,109]. In Table 5, they are listed curing times and temperatures recommended by the manufacturer.

Table 5. *Recommended curing times and temperatures to fabricate PDMS* [49].

Temperature (°C)	Time
25	48 hours
100	35 minutes
125	20 minutes
150	10 minutes

The monomer and the curing agent can be mixed at a different ratio besides the 10:1 [14] and, as consequence, some properties change, namely, mechanical [110],

optical [111] and gas permeability [112]. Mixing at a ratio that implies more cure agent, results in a faster hardening time, in a less sticky cured PDMS and in a more fragile PDMS sample. In contrast, mixing with less cure agent, results in a longer hardening time, in a stickier cured PDMS and in better mechanical properties. Khanafer et al. [110] found that Elastic Modulus increases as the mixing ratios increase up to 9:1, after which the Elastic Modulus starts to decrease as the mixing ratio continues to increase.

3.2 PDMS characterization techniques

To investigate if PDMS is a suitable material for masks, the most important tests to be performed are goniometry, transmittance and tensile test.

3.2.1 Goniometry testing

Wettability assay is accomplished using a goniometer that allows WCA measurement. The goniometer used in this assay was the optical contact angle 25 (OCA 25) and it is presented in Figure 5. The contact angle measures the free energy on a solid surface. This technique analyses the shape of a sessile drop on a solid surface and provides information about the contact angle formed between the solid and the tangent of the drop's surface [113]. This test allows to characterize PDMS as hydrophobic or hydrophilic, which is an important property for the mask. WHO [15] recommends that the innermost layer of a mask should be hydrophilic in order to absorb the expelled breathing droplets, preventing them to be released to the surrounding environment and possibly infect other people. On the other hand, the outermost layer should be hydrophobic, with the purpose of repel possibly infected droplets in the air. Water droplets bead up when in contact with a hydrophobic layer, which means that infected droplets will have more difficulty to attach the mask.

The sessile drop method is performed by placing a water drop on the surface of PDMS. The drop can be observed through a lens and the contact angle is measured with a goniometer [113]. This angle is influenced by the surface energy of the sample and by the superficial tension of the liquid. In hydrophobic surfaces, high values of WCA are observed and the surface is characterized for having poor wettability, a weak adhesion and low surface energy. In this surface, the drop spreads partially. On the other hand, on a hydrophilic surface,

the drop spreads over the entire surface, presenting lower WCA. This means that the surface has good wettability, good adhesion, and a higher surface energy [114].

This method presents an important limitation. Drawing the tangent line of the drop to measure the contact angle is a factor that limits the reproducibility of angles. This technique relies on the precision of the user when drawing the line, leading to incorrected measures. It is possible to use computer analysing. However, there are also errors associated to the definition of the tangent.

To conduct the goniometry test on PDMS, firstly, a PDMS sample was placed on the equipment and aligned with the measurement system. Then, the micro syringe was lowered, and a water drop (0.3 µL) was placed in the PDMS surface. An image of the drop on the PDMS surface is presented in Figure 6. The image is sent to a software that draws the tangent line and the water contact is measured. To minimize the errors, five measurements per sample were performed and an average of the results was calculated.

The WCA was measured on PDMS samples fabricated with different mixing ratios (5:1, 10:1 and 20:1) and with different percentages of recycled PDMS (10%, 20% and 30%).

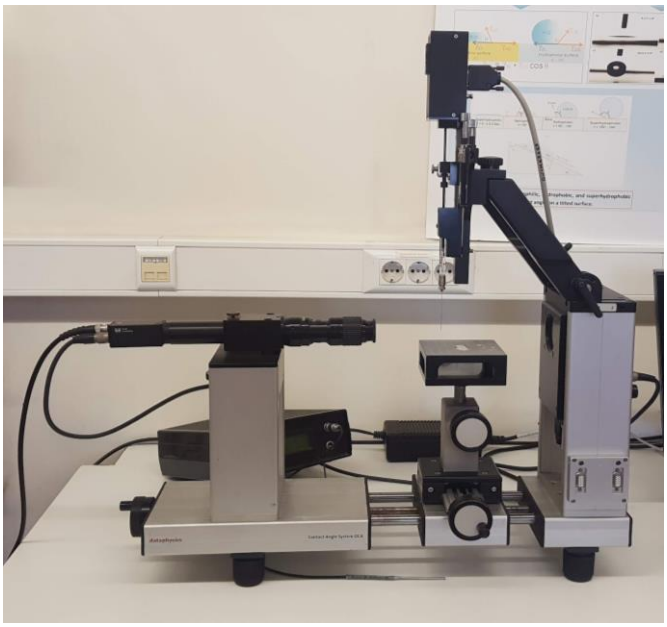


***Figure 5**. Goniometer used to perform wettability tests.*

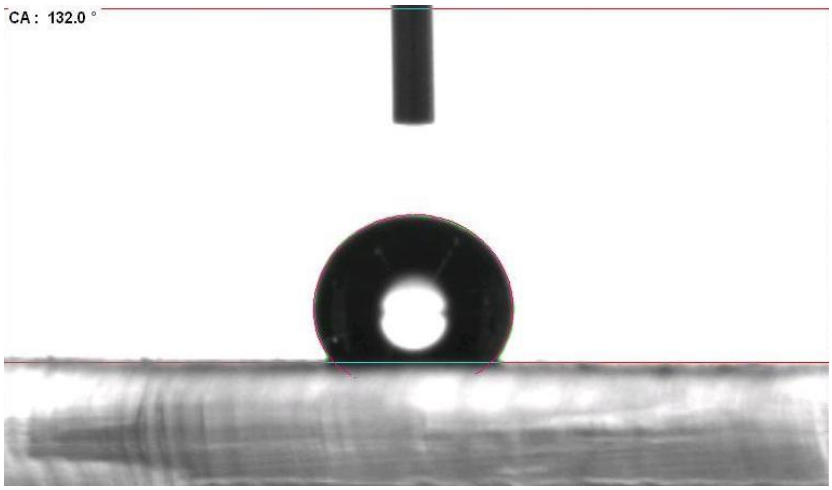


Figure 6. *Microscopic image of a water droplet for goniometry measurement of PDMS 20:1 sample.*

3.2.2 Transmittance measurements

Transparency is another important property for a mask that is meant to be transparent. A transmittance assay should be performed in order to quantify the "transparency" of PDMS. A spectrophotometer is the equipment used to perform this assay that measures the transmittance in UV, visible and infrared (IV) regions of the electromagnetic spectrum. This equipment consists of a light source, a monochromator, and a detector. Usually, the light source has two types of lights, one of deuterium or xenon which emits electromagnetic radiation in UV region, and the other of tungsten which is used for wavelengths in the visible region. The monochromator consists of a diffraction network that allows to split the beam in all its wavelengths. The radiation passes through the sample and reaches the detector, which reads the intensity of the transmitted light.

Transmittance (T) is the ratio between the measured light (I) and the incident light (I_0), and can be expressed as a percentage:

$$\mathrm{T} = \frac{\mathrm{I}}{\mathrm{I}_0} \qquad \textit{(Equation 1)}$$

Transmittance was conducted in a UV-2600 spectrophotometer showed in Figure 7. The assay was conducted sweeping the spectrum from 200 nm to 800 nm, every 1 nm.

Figure 7. *Spectrophotometer used for the transmittance tests. The yellow piece is an adapter used to place the PDMS sample in the equipment.*

A material is considered transparent to visible light if the transmittance to a light range between 380 and 780 nm (visible light) [115] is approximately 100%. Cruz-Félix et al. [116] demonstrated that PDMS transmittance depends on the curing temperature and the ratio of which it is prepared. Figure 8 shows the results obtained for five different mixture ratios of PDMS (M1 is 10:1, M2 is 10:1.25, M3 is 10:1.5, M4 is 10:1.75 and M5 is 10:2).

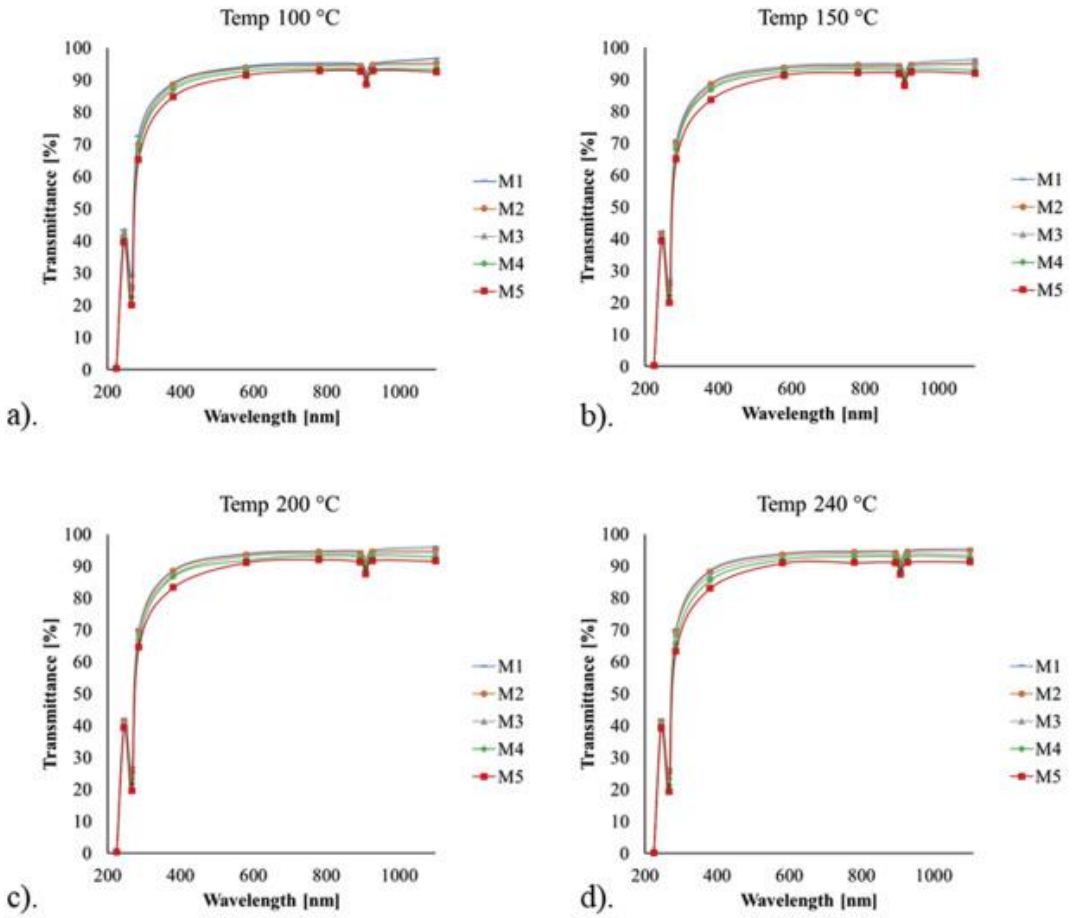


Figure 8. *Transmittance spectra of PDMS samples cured at a)100 °C, b) 150 °C, c) 200 °C and d) 240°C* [116].

The results obtained in this study demonstrate that an increase of both, ratio and curing temperature produces a slight decrease of transmittance in the visible spectral range.

3.2.3 Mechanical Testing

Considering that face masks are susceptible to mechanical actions, it is important that the mask is made with materials capable of bearing these actions. The main mechanical force to which the mask is susceptible is traction. PDMS is an elastomer which means that it is a viscoelastic material. Mechanical tests can be of two types: static tests and dynamic tests. Static testing is performed at low frequencies (less than 1 Hz) while dynamic test is performed under higher frequencies [117]. To study a polymer that is meant to be subjected to vibration, shock or impact, a dynamical test is crucial to understand its behaviour under these circumstances [117]. From a dynamic mechanical test, it is possible to obtain the curve Storage or Elastic Modulus (E) versus temperature, frequency, or strain, which will help to understand if a PDMS mask can bear a daily basis use. Additionally, a dynamic test should be

performed alongside with a static test because, only that way, it is possible to achieve more accurate results [117]. Therefore, a static mechanical assay was also performed to obtain a strain versus stress curve. The Hooke's Law (Equation 2) states that Elastic Modulus (E) is the ratio between tension (σ) and strain (ε) [118]:

$$E = \frac{\Delta\sigma}{\Delta\varepsilon} \qquad \textit{(Equation 2)}$$

Elastic Modulus can be affected by treatments that may be applied to the PDMS, by the curing temperature and the time, and by the mixing ratio used to fabricate the PDMS samples [110,119]. A flexible material has a low Elastic Modulus, so, it is expected that the PDMS has a lower Elastic Modulus when compared with other stiffest materials, like PC (polycarbonate). Based on the literature, Elastic Modulus of PC is around 1.45±0.11 GPa [120], so it is expected a much lower value for PDMS, in the order of MPa. Wang et al. [119] performed compression dynamic tests on PDMS and achieved an Elastic Modulus of 3.59 MPa for 5:1 PDMS and 2.66 MPa for 10:1 PDMS. It is important to notice that the Elastic Modulus measurement strongly depends on the tests parameters, on the used method for the assay and on the fabrication conditions of the sample.

In order to perform the mechanical assays, PDMS samples were prepared with different mixing ratios (5:1, 10:1 and 20:1) and with different percentages of recycled PDMS (10%, 20% and 30%). An aluminium mould (Figure 9) was used to produce samples with 30 mm length, 6 mm width and 2 mm depth. The DMA (Dynamical Mechanical Analysis) equipment used to carry out this assay is presented in Figure 10.

***Figure 9**. Aluminium mould to fabricate PDMS samples for mechanical testing.*

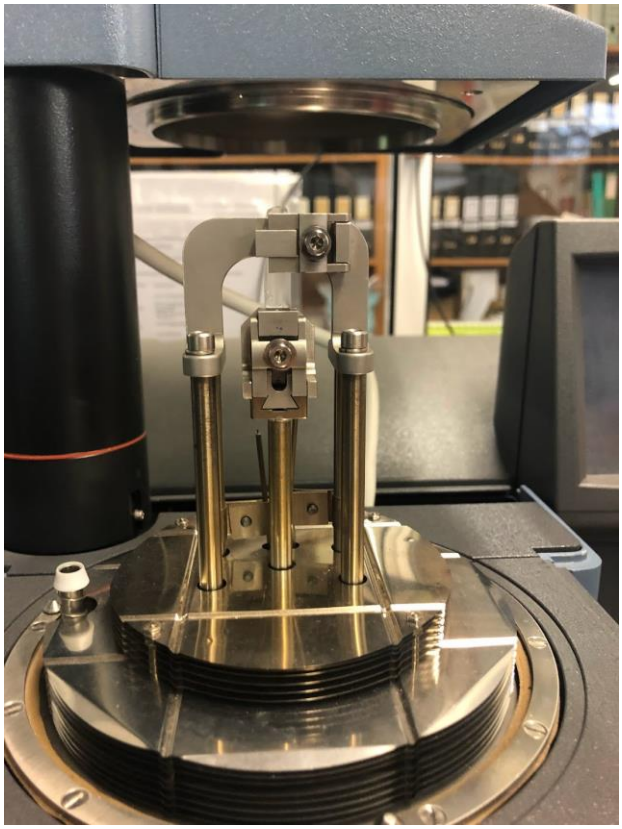


***Figure 10.** DMA equipment used to perform mechanical assays.*

3.2.4 Other tests

In addition to the mentioned tests, other tests are commonly performed, namely, scanning electron microscopy (SEM), gravimetry, nanoindentation, tensile test, X-Ray photoelectron spectroscopy (XPS) and Fourier transform infrared spectroscopy (FTIR) [25]:

- SEM allows morphological characterization of PDMS samples [45,121–124].

- Gravimetry is a method based on gravitational techniques to quantify changes in PDMS sample weight. For example, this method is useful when it is needed to verify if there was or not degradation of PDMS after chemical immersion [125].
- Nanoindentation offers the possibility of studying mechanical properties of the outermost layer of PDMS, which is susceptible to destruction due to different treatments, such as UV irradiation [126].
- XPS is a technique based on the photoelectric effect that allows identification of the elemental composition of the material. This method is useful when it is needed to verify if any changes in surface composition occurred after PDMS receives any treatment [127–129].
- FTIR is a method used to obtain the infrared spectrum of absorption or transmission of the PDMS sample. This technique allows examination of some treatment effects on the cross-linking of PDMS [123,128,130].

3.3 Development of PDMS samples for inclusion in transparent face masks

3.3.1 Native PDMS

PDMS samples fabricated with different ratios were characterized in terms of wettability, transparency and mechanical tests. Mixing ratio of 10:1 (w/w) is the recommended ratio by the manufacturers. However, it is possible to achieve some different properties when using different mixing ratios. Therefore, three different ratios were prepared: 5:1, 10:1 and 20:1, in order to choose the best one to accomplish the goal of making a transparent face mask. The samples used for transparency tests were the same as those used for wettability tests. The preparation of the samples for mechanical tests followed the same procedures as the wettability tests, except for the used mould. Rectangular shaped PDMS samples with 60 mm length, 30 mm width and 2 mm depth were used to conduct wettability and transmittance tests. For the mechanical tests an aluminium mould with 30 mm length, 6 mm width and 2 mm depth was used. The preparation of the samples followed the following procedures:

1. Sylgard 184 silicone elastomer was used. The silicone base and the curing agent were mixed with the respective mixing ratio (w/w) in a cup using a spoon.

2. After mixing, the cup is placed inside a desiccator to remove the bubbles. The time for the bubbles' removal depends on the power of the equipment. The equipment used in this project allowed removing the bubbles in about 20 min.
3. PDMS is then poured onto an acrylic mould for wettability and transmittance tests and onto an aluminium mould for the mechanical test. The moulds were placed in the oven at 80 °C. The curing time depends on the mixing ratio; 5:1 sample took around 1 h, 10:1 samples around 1h and 30 min and 20:1 around 2 h and 30 min to cure.
4. The final step consists of removing the cured PDMS from the mould.

3.3.2 Recycled PDMS

The possibility of recycling the PDMS at the end of life or after its use, was also studied. For that, the masks are grated in small solid particles of PDMS, being posteriorly incorporated in the original liquid PDMS (before curing). In Figure 11 it can be observed the PDMS grinded by a hand blender and by a manual food grater. The food grater allowed to obtain substantially smaller PDMS particles being, as a consequence, the chosen method to grate the PDMS. In addition, when available, automatic grinded equipment could also be used.

The grated PDMS can be mixed with the liquid silicone base and curing agent before cure. In order to investigate the percentage of PDMS grains that can be added to native PDMS without significantly change its properties, three different percentages (w/w) were tested: 10%, 20% and 30%.

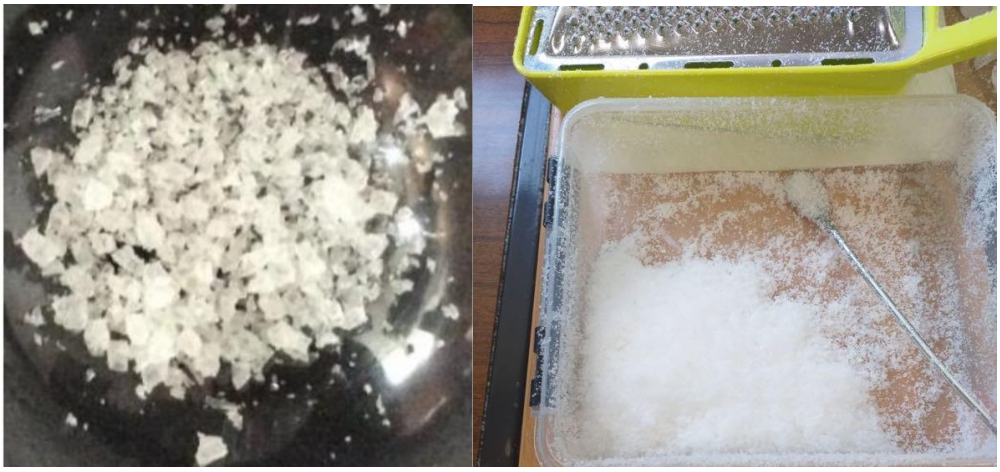


Figure 11. *(a) PDMS grated with a hand blender and (b) with a manual food grater.*

The preparation of the recycled samples to perform the characterization tests, followed the same procedures as the native PDMS, with the exception of mixing the PDMS grains in the first step of the preparation. When mixing the grains with the liquid PDMS, it can be hard to obtain a homogenous mixture. PDMS grains tended to agglomerate instead of dispersing homogenously in the mixture. This event must be taken in consideration when PDMS is poured onto the mould. This means that the samples may not have the exact percentage of grains when compared with their preparation.

3.4 Design and fabrication of a face mask comprising a combination of PDMS with fabric

Based on the characterization of the native PDMS and the recycled PDMS, it was concluded that a mask made only with PDMS is not feasible. Analogously to what happens in a surgical mask, breathing causes condensation on PDMS. Condensation is characterized by the formation of water bubbles in the surface of the material and that would affect PDMS transparency. Additionally, the standard for the certification of respirators imposes a minimum breathability value and PDMS has a null breathability. Developing a fully transparent PDMS mask would involve high technology to overcome these limitations. Therefore, it was investigated the combination of PDMS and fabric. The idea of fabricating a mask with fabric and a PDMS window in the mouth region emerged in PDMSmask4ALL project. The mask was developed taking in consideration the community face coverings guide CWA 17553:2020 [131]. This guide provides information about the minimum requirements, assay methods and ways of using facial communitarian masks.

Initially, a design for the mask needs to be chosen. The design must allow the incorporation of the PDMS window, without affect the comfort of the mask and respecting the certification requirements. The mask comprises an air permeable filtering portion and a transparent central window, wherein the air permeable filtering portion is formed from a fabric composed by an outermost layer of interlock 100% recyclable polyester; an intermediate layer of 100% polyester, preferably recyclable polyester, and an innermost layer of VOILE organic cotton, and the transparent portion is formed by a PDMS film covering the nose and mouth. In this mask, the transparent portion is air impermeable. The transparent portion includes along its periphery a small contour of fabric that is later on joint to the air

permeable fabric in its outer side edges by using a sewing process. The transparent film has a horizontal width that is greatest than vertical length.

Two types of masks were fabricated: one with a native PDMS window and another with a recycled PDMS window. According to the results presented in section 4.1.1, the PDMS 5:1 mixing ratio was the one that outputted better performance and, consequently, was the chosen for being applied on the face mask. For the recycling, the same was applied, thus the recycling process was performed only with this ratio. The fabrication process of the mask with native PDMS window is detailed in Figure 12 and in Figure 13a) a mask prototype can be observed. One of the requirements for mask certification is that the mask must not be sewn and if they are, the stitches must be sealed. For that reason, all the stitches in the mask were sealed with PDMS and, posteriorly, cured in the oven at 80 °C for 15 min. This ensures that no air, and consequently, no virus passes through those stitches. A representation of this sealing is presented in Figure 13b). That way, it is possible to obtain the required transparency and breathability is guaranteed by the surrounding fabric. Both components can filter the virus.

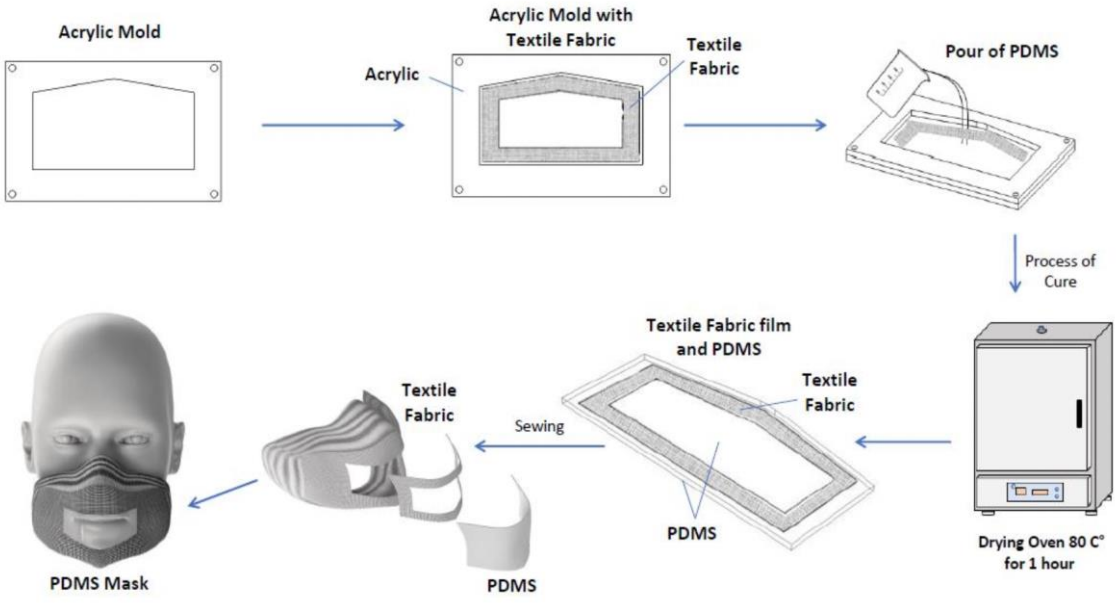


***Figure* 12.** *Schematic representation of process for fabricating the facial mask with PDMS and fabric.*

(a)

(b)

Figure 13. *(a) Mask prototype with fabric and PDMS. PDMS was fabricated with 5:1 ratio PDMS and (b) the stitches sealed with PDMS on the innermost layer.*

An acrylic mould (Figure 14) with the desired window design was fabricated to produce the PDMS films. The liquid PDMS was poured by weight in order to ensure that all PDMS

windows had the same thickness. That way, 11 g of PDMS was poured onto the acrylic mould and after curing, PDMS windows with 2 mm thickness were accomplished.

Additionally, in order to fabricate a recyclable mask, recycled PDMS windows were prepared. The manufacturer process for recyclable masks is the same as for masks with native PDMS, but instead of placing a native PDMS window, it was placed a recycled PDMS window. According to the results presented in the section 4.1.2, the 10% of recycling was the one that outputted better performance and, consequently, was the chosen percentage for being applied on the recycled face mask. Since the integration of recycled PDMS leads to an increase of the fragility of the window, it was decided to fabricate thicker windows than those of native PDMS. Therefore, 12.6 g of liquid PDMS were mixed with 1.4 g of recycled PDMS grains, to complete 14 g in total. After curing, the windows were 2.5 mm thick. This process is enlightened in Figure 15 and an image of the recyclable PDMS mask is presented in Figure 16.

Figure 14. *Acrylic mould to produce the PDMS films for the mask.*

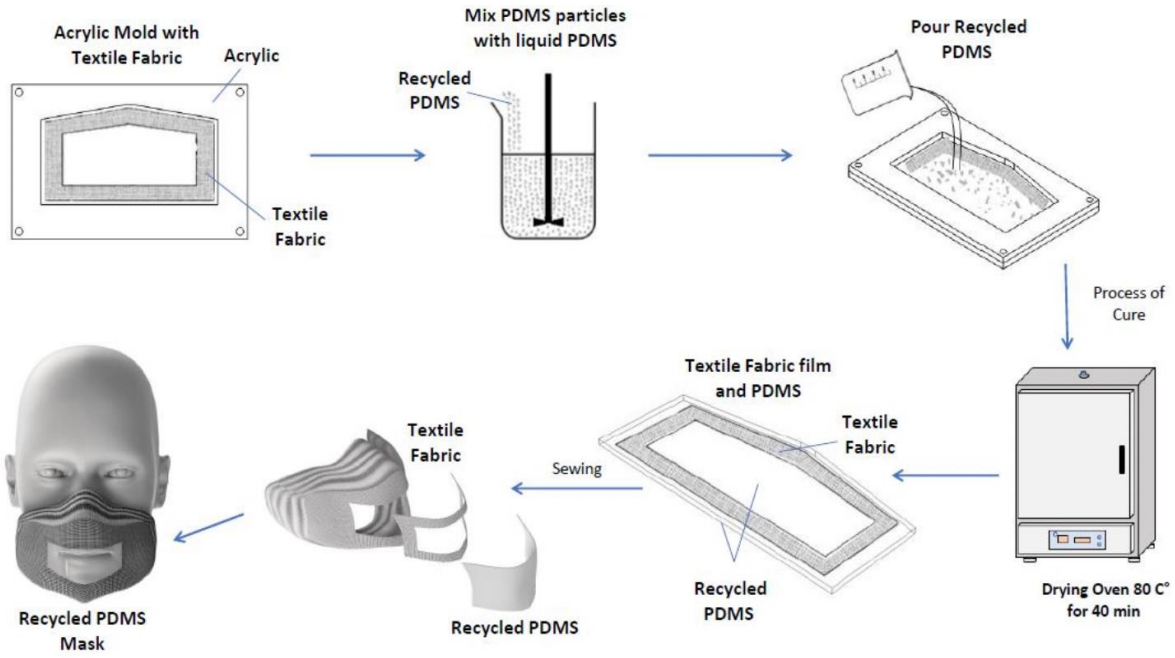


Figure 15. *Schematic representation of process for fabricating the facial mask with recycled PDMS and fabric.*

Figure 16*. Mask prototype with fabric and PDMS made with a 5:1 mixing ratio and a 10% of recycled PDMS.*

4. RESULTS AND DISCUSSION

In this chapter, the results achieved from the experimental characterization of PDMS are presented and a critical analysis is made. Furthermore, a comparative study about wettability, transmittance and tensile strength between recycled and native PDMS is also presented. Additionally, the characterization of several fabrics to include in the face mask are presented and the selection of the most suitable is discussed. Finally, the results of the mask certification are showed and analysed.

4.1 PDMS characterization

In order to characterize the properties of PDMS to assess its viability for application in face masks, three tests were performed: goniometry, transmittance and tensile strength test. The methodologies used to perform each test are described in the section 3.

Wettability assay was accomplished using a goniometer that allows contact angle measurement. A spectrophotometer was used to perform the transparency tests, applying a light range between 200 and 800 nm. To conduct the mechanical assay, a DMA equipment was used. Two types of assays were performed: a dynamic and a static assay. Dynamic assay was conducted by frequency sweep method and static assay allowed drawing stress/strain curve. Both assays provide information about the Elastic Modulus of PDMS, being that the static assay provides a complementary information for the dynamic assay.

4.1.1 Native PDMS

Goniometry

To conduct the goniometry test, five measurements of the WCA per sample were performed in a goniometer (Figure 5) and an average of the results was calculated. This test allows the characterization of PDMS as hydrophobic or hydrophilic, which is an important property for the mask. Wettability assay's results are presented in Table 6.

***Table 6**. Goniometry test results for different PDMS mixing ratios.*

PDMS samples	5:1	10:1	20:1
Contact Angle (°)	143.7±5.7	136.5±7.2	135.5±3.8

A hydrophobic surface is characterized for having WCA between 90° and 150°. Observing Table 6, it can be stated that PDMS is hydrophobic independently of the ratio, because their contact angles are between 135° and 144°. Additionally, it can be observed that the higher the ratio, the lower is the contact angle.

Despite being hydrophobic, that does not mean that mask condensation can be avoided. That will mostly happen in super hydrophobic material, as mentioned in sub-section 2.4, which are characterized by having contact angles above 150°. The 5:1 mixing ratio samples were the ones that presented a closer behaviour to a super hydrophobic surface. Developing a mask with super hydrophobic characteristics can be very useful, since it will provide an antibacterial feature to the mask.

Transparency

Transparency is another important property for a mask that is meant to be transparent. For that reason, transmittance assay was performed on the different mixing ratio samples. The spectrophotometer presented in Figure 7 was used to perform the transparency tests. Transmittance curves of the different samples are presented in Figure 17.

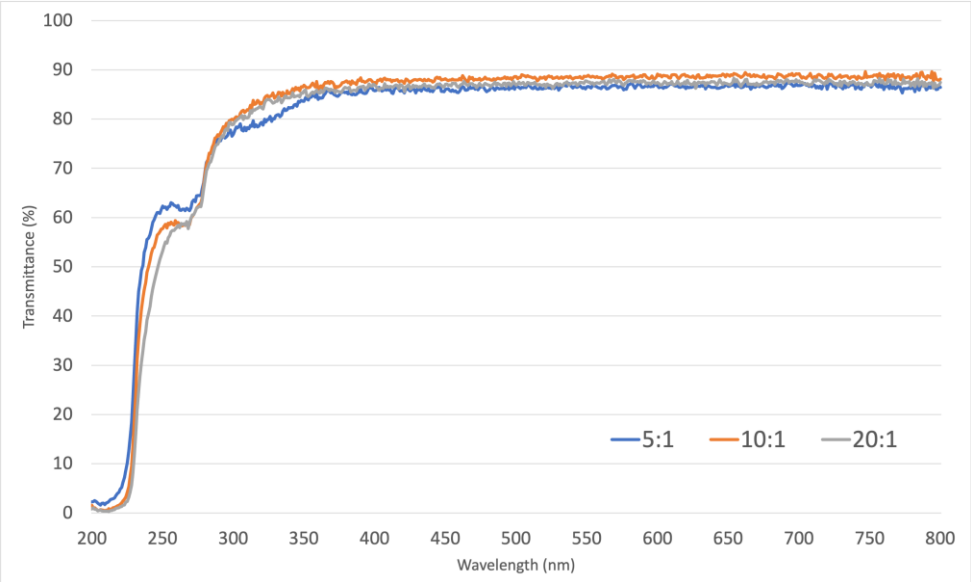


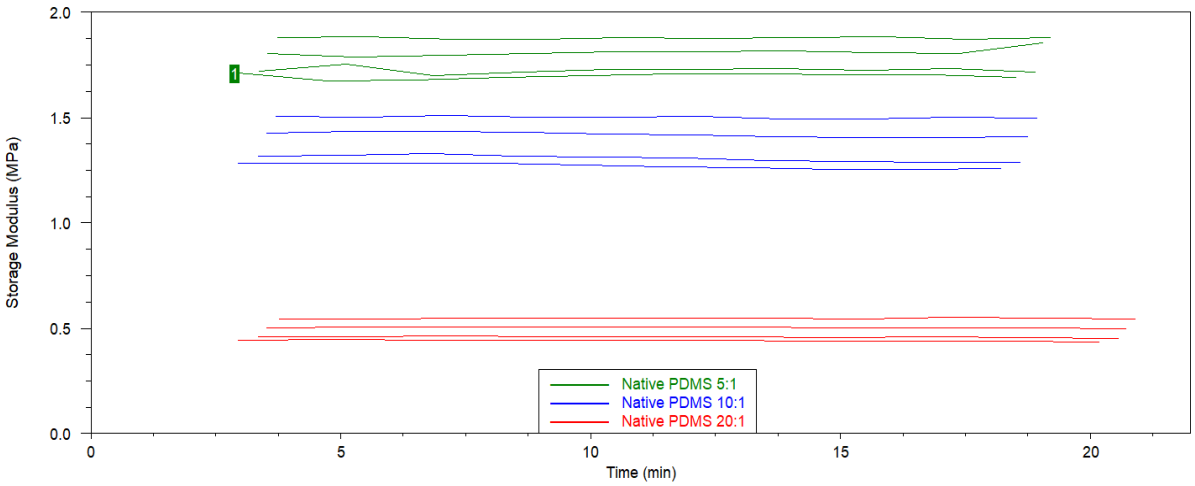
Figure 17. *Transmittance curves for PDMS 5:1, 10:1 and 20:1 mixing ratio samples.*

From the transmittance curves it can be stated that the mixing ratio does not cause significant changes in the transparency of the sample. It can also be stated that PDMS is highly transparent at the visible light (380–780 nm), achieving almost 90% of transmittance. Additionally, it can be observed that 10:1 sample has the higher transmittance and that the

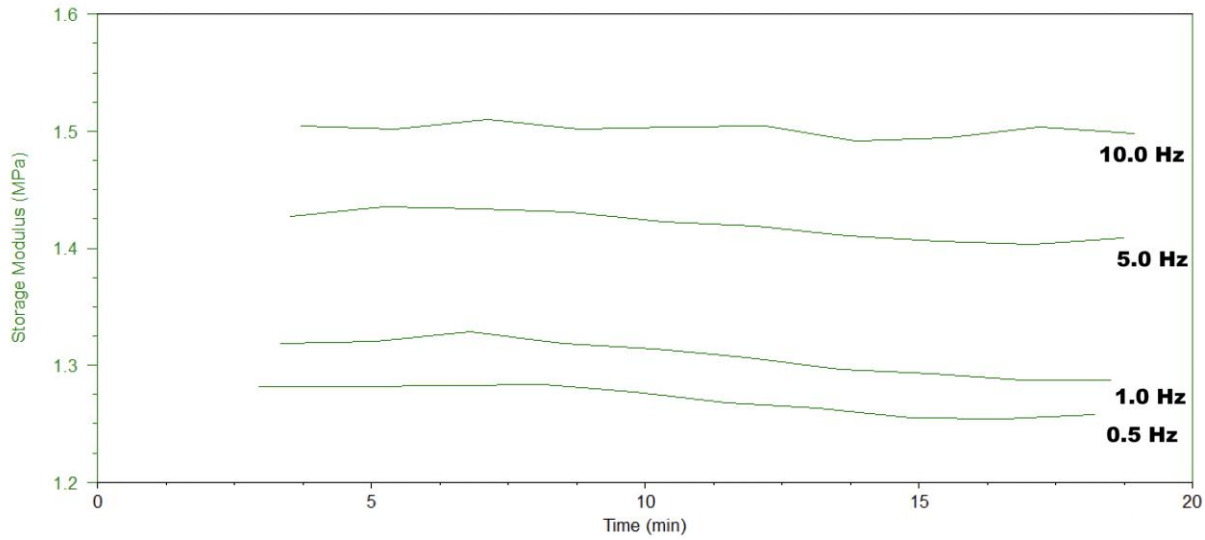
5:1 has the lower, for the visible spectral range. As the wavelength decreases, the transmittance exponentially approximates to 0%, which means that PDMS offers some UV protection, and this could be an interesting property since the goal is to make a PDMS mask that is meant to be used outside.

Mechanical assays

Tensile strength tests were conducted on PDMS using the DMA equipment of Figure 10. Because PDMS is a viscoelastic material, a dynamic test is recommended to calculate the Elastic Modulus. The results of the dynamic test are presented in Figure 18.



(a)



(b)

Figure 18. *Tensile test on rectangular shaped PDMS samples with size 13.6 x 6.1 x 2.1 mm (L x W x T). Frequency sweep was performed for 0.5, 1.0, 5.0 and 10.0 Hz. (a) Storage Modulus results for PDMS samples with mixing ratios of 5:1, 10:1 and 20:1 and a (b) close up curve for PDMS 10:1 sample. To note that for each of the samples, from the bottom to the top, each of the curves represents, respectively, 0.5, 1.0, 5.0 and 10.0 Hz.*

By Figure 18, it can be stated that Storage Modulus or Elastic Modulus depends on the mixing ratio of PDMS. The higher the mixing ratio, the lower is the Elastic Modulus, the more elastic is the sample and, therefore, the more it can handle tension strengths. Additionally, it can be observed that for each sample, the Elastic Modulus depends on the applied frequency. The higher the frequency, the higher is the Elastic Modulus, which means that PDMS loses capability of outstanding traction with increasing the frequency. For example, for a frequency of 1.0 Hz, the obtained Elastic Modulus were, approximately, 1.6 MPa for 5:1 sample, 1.2 MPa for 10:1 sample and 0.4 MPa for 20:1 sample.

In Figure 19, the results for the static assay are presented. The Elastic Modulus were calculated using Equation 2, which is the same as calculating the slope for each curve. The results are presented in Table 7.

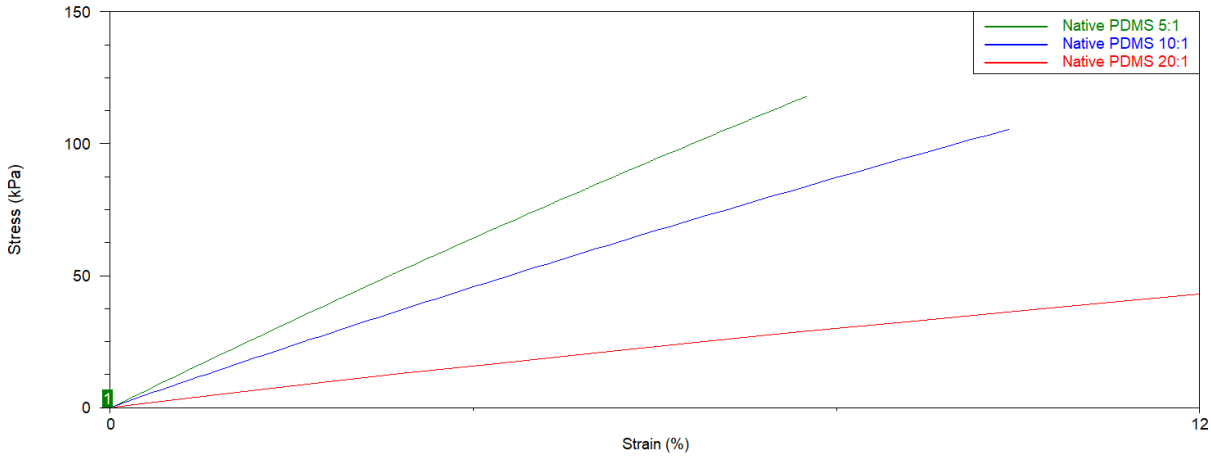


***Figure 19**. Stress vs strain curve for 5:1, 10:1 and 20:1 mixing ratio PDMS samples.*

***Table 7**. Elastic Modulus results based on the static assay and the Hooke's law for 5:1, 10:1 and 20:1 mixing ratio PDMS samples.*

PDMS samples	5:1	10:1	20:1
E (Elastic Modulus, MPa)	1.52	1.04	0.39

Table 7 allows to confirm the previous results, since the results of static assay were similar to those obtained by the dynamic assay, as expected. These results are lower than those obtained by Wang et al. [132] and that can be explained by the fact that were performed different types of test: Wang et al. performed a compression test and in this work

a tension test was performed. The authors obtained the following Elastic Modulus values: 3.59±0.11 MPa for 5:1 sample and 2.61±0.02 MPa for 10:1 sample. Additionally, the cure conditions of the PDMS were slightly different: Wang et al. cured the samples at 65 °C for 12 h and in this work the samples were cured at 80 °C for 1.5 h.

In conclusion, those results showed that the samples prepared with 5:1 mixing ratio presented a higher contact angle, approaching a super hydrophobic behaviour, which can be an advantage from the perspective of fabricating an antibacterial surface. Additionally, it was proved that the sample with lower Elastic Modulus was the 5:1 ratio, which means that is the less elastic but also the less sticky sample. Finally, the transmittance test showed that are no significant differences between samples prepared with different mixing ratios. For these reasons, the 5:1 ratio was chosen to be applied on the mask.

4.1.2 Recycled PDMS

The tests performed to characterize native PDMS were repeated for the recycled PDMS, with the purpose of investigate if the recycling method developed was adequate. Goniometry, transmittance, and mechanical assays were conducted in the same conditions as the native PDMS. A comparison between the results of native and recycled PDMS was made.

Wettability

To obtain the recycled samples, grains of solid PDMS are mixed with liquid PDMS. Since the thickness of the sample is thin, it is expected that with the increase in the percentage of recycled PDMS, the surface becomes more and more rough, due to the increase in the quantity of grains. This means that an increase in the percentage of recycled PDMS will lead to an increase of the sample hydrophilicity. The measured WCA are presented in Table 8.

***Table 8**. Goniometry test result for different percentages of recycled PDMS.*

PDMS samples	Native PDMS 5:1	10% recycled PDMS 5:1	20% recycled PDMS 5:1	30% recycled PDMS 5:1
Contact Angle (°)	143.7±5.7	138.2±4.2	136.8±5.7	129.6±4.8

Observing Table 8, it can be stated that PDMS is hydrophobic independently of the percentage of recycled PDMS, because their contact angles are between 129° and 139°. In comparison with native PDMS, it can be stated that recycling PDMS decreases its hydrophobicity. Additionally, it can be observed that the higher recycled PDMS percentage, the lower is the contact angle, due to the increase of the surface roughness. However, these results do not interfere with fabrication of a mask with antibacterial properties, since there are several ways to reach that feature in addition to super hydrophobic coating.

Transparency

It is expected that the addition of PDMS grains in the liquid will lead to a decrease of PDMS transparency, because of the agglomeration of the grains [70]. In Figure 20 the transmittance curves of the different samples are presented.

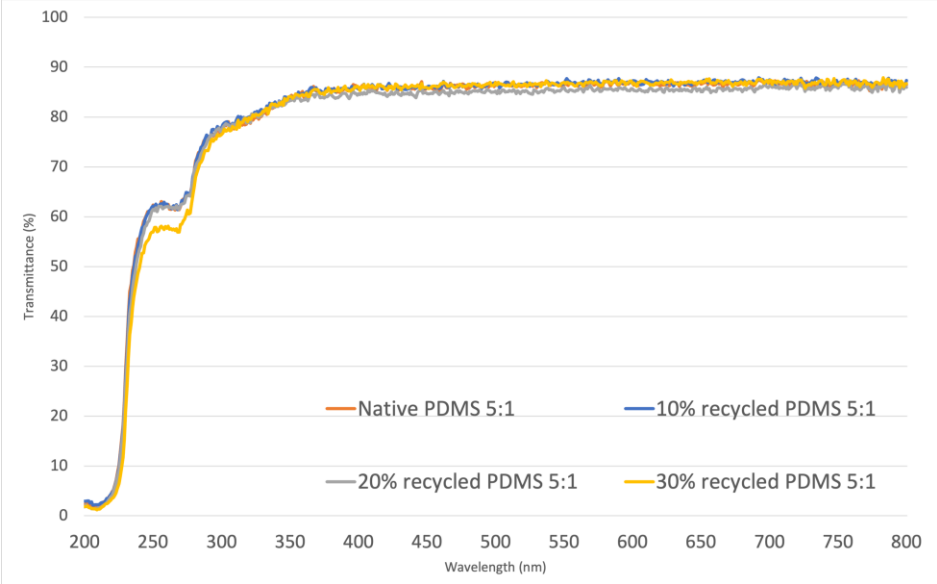


***Figure 20**. Transmittance curves for PDMS samples with 10%, 20% and 30% recycled PDMS.*

The transmittance results for the recycled PDMS are very similar to the native PDMS. Despite the agglomeration of the recycled grains, a decrease on the transmittance was not observed. Additionally, as the native PDMS, recycled PDMS is, as well, highly transparent to the visible light with a transmittance value of approximately 90%.

Mechanical assays

It is expected that recycling PDMS will lead to an increase of the Elastic Modulus. Mixing recycle PDMS grains in liquid PDMS will make it more fragile after curing. Figure 21 presents the curves of the dynamic test.

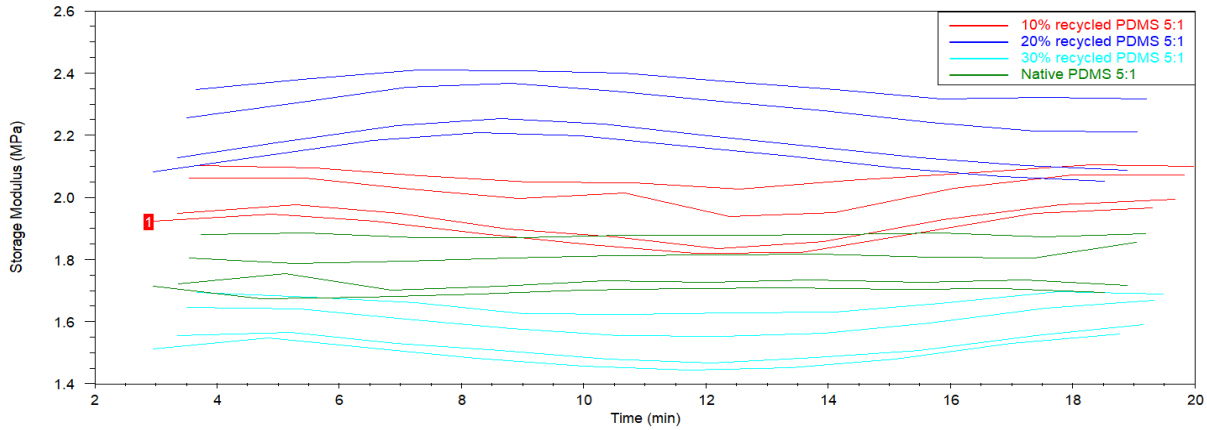


Figure 21. *Storage Modulus results for 10%, 20% and 30% recycled PDMS samples. PDMS samples have a rectangular geometry with size 13.6 x 6.1 x 2.1 mm (L x W x T). Frequency sweep was performed for 0.5, 1.0, 5.0 and 10.0 Hz. To note that for each of the samples, from the bottom to the top, each of the curves represents, respectively, 0.5, 1.0, 5.0 and 10.0 Hz.*

The integration of the PDMS grains makes PDMS more fragile and, consequently, less elastic. By Figure 21 it can be stated that Elastic Modulus depends on the percentage of recycled PDMS. Additionally, and similarly to native PDMS, the Elastic Modulus increases with increasing the applied frequency. In comparison to the native PDMS, Elastic Modulus by percentage of recycled PDMS for 1.0 Hz were, approximately, 1.9 MPa for 10%, 2.15 MPa for 20% and 1.3 MPa for 30%. The Elastic Modulus obtained for 30% recycled PDMS was not the expected and that can be explained by the difficulty of obtaining a homogenous mixture of the PDMS grains in the liquid PDMS.

Stress versus strain curve is presented in Figure 22. The respective slopes (Elastic Modulus) were calculated and are presented in Table 9.

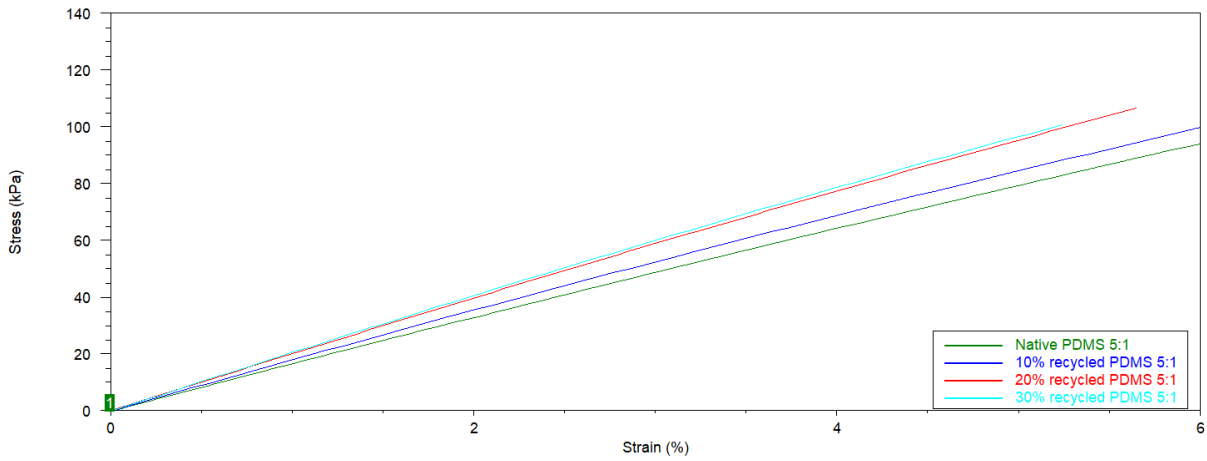


Figure 22. *Stress vs strain curve for 5:1 mixing ratio PDMS with 10%, 20% and 30% of recycled PDMS.*

Table 9. *Elastic Modulus calculation based on the static assay and on Hooke's law for 10%, 20% and 30% recycled PDMS.*

PDMS samples	Native PDMS 5:1	10% recycled PDMS 5:1	20% recycled PDMS 5:1	30% recycled PDMS 5:1
E (Elastic Modulus, MPa)	1.52	1.74	1.98	2.02

The results of Table 9 confirm the theory that the Elastic Modulus increases as the recycled PDMS percentage increases.

In conclusion, the characterization tests of recycled PDMS showed that an increase of recycled PDMS results in a decrease of the hydrophobicity. As for the transmittance tests, no significant changes were found. However, an increase on the percentage of recycled PDMS can lead to a decrease of the transparency of the film. Moreover, the mechanical tests showed that an increase on the recycled PDMS results in an increase of the Elastic Modulus, which means that the samples become more fragile. Based on these results, 10% was the percentage of recycling chosen to integrate the mask.

4.2 Fabric characterization

Choosing the fabric for the mask has to be made accordingly to the standards, so that the breathability and filtration are adequate to the desired mask level. It is imposed by the standard that in the breathability test a minimum air flow of 8 L/min must be guaranteed. The breathability test for certification is performed on the mask in a 4.9 cm^2 region with a 40 Pa pressure. A FX-3300 air permeability tester, presented in Figure 23, was used to measure the fabric breathability. This equipment is similar to that used in certification test, but showing the air permeability in $L/m^2/s$, with a pressure of 40 Pa and in a 20 cm^2 region. Measurements made for 6 types of fabrics are presented in Table 10. Each fabric was submitted to three readings and an average of the values was calculated. The result, in $L/m^2/s$, was converted to L/min, for an area of 20 cm^2.

Figure 23. *Equipment used to perform the permeability tests in the fabrics.*

Several types of fabric were acquired and developed. For example, Otojal developed a three-layer fabric (fabric number 6 in Table 10), which was certified for a level 2 mask. That was the chosen fabric, and it is compound as follows: for the outermost layer an INTERLOCK type fabric with 125 g/m^2 density, for the intermediate layer an interfacial fabric with 58 g/m^2 and for the innermost layer a Voile type fabric with 75 g/m^2. The outermost layer has hydrophobic properties, the intermediate layer has filtration features, and the innermost layer is 100% organic cotton, capable of absorbing the water bubbles that result from breathing.

Table 10*. Air permeability measurements.*

Fabric	Air permeability ± SD (L/m^2/s)	Pressure (Pa)	Air flow (L/min)
Fabric 1 (2 plies)	318±2	40	38.1
Fabric 2	104±8	40	12.4
Fabric 3	103±1	40	12.4
Fabric 4	183±7	40	22.0
Fabric 5	220±10	40	26.4
Fabric 6	250±13	40	30.0

The results of Table 10 allowed to conclude that all the tested fabrics meet the minimum breathability requirements, since all of them presented an air flow higher than 8 L/min for a 40 Pa pressure. However, other aspects must be taken in consideration. It must be chosen a fabric with a safety margin of air permeability in order to guarantee that it will pass the certification tests. Additionally, the mask will not only be made with fabric, but it also has a PDMS window with null breathability. Again, Table 10 allowed to conclude that fabric 6, the chosen fabric, meets the minimum breathability requirements with a good safety margin. Additionally, although the chosen fabric is not the one with the greatest breathability, it is the only that is recyclable and, therefore, the only one that allows to fulfil the objective of manufacturing a recyclable mask.

4.3 Preliminary validation of the mask with PDMS and fabric

Before sending the masks to the certification laboratory, some tests were performed in order to verify if they met the requirements of the certification. CWA 17553:2020 guide [131] requires that community face coverings specified as reusable shall withstand at least 5 cleaning cycles with a minimum washing temperature of 60 °C. If any damage to the mask is detected after each cleaning cycle, the mask is deemed non-compliant.

To ensure that the developed masks passed the cleaning assay, qualitative assays were performed in a normal washing machine. The masks were washed for 5 cycles at 60 °C, for 30 min. After each cycle, no significant changes were observed on PDMS and no general damage, such as tears, detachment of the head harness, less accurate fit, deformation, or wear, was detected.

Additionally, it was taken in consideration the way that breathability tests are performed by the certification laboratory. That information comes in CWA 17553:2020 guide [131], which states that the test is performed in five regions of the mask with an area of 4.9 cm^2. The regions are top left and right, bottom left and right, and centre. The regions should be adjusted in such way that the measurements are not made on top of a sewed region. Based on this, the mask design was carefully adjusted in order to not interfere with the measurements. The main adjustment that had to be done was the placement of the PDMS window. The design was adjusted so that the central region of the mask was fabric and not PDMS. The final design can be observed in Figures 13a) and 16.

At this point, the projected masks are ready to be certified. In the next sub-section, the results of the certification are presented.

4.4 Mask certification

The masks were certified by Equilibrium which is a laboratory that is accredited by Portuguese Institute of Accreditation (IPAC) according to standard NP EN ISO/IEC 17025. This laboratory performs assays and evaluation based on CWA 17553:2020 guide [131]. The evaluation englobes visual inspection of the mask, evaluation of their reuse and cleaning cycles, analysis of antibacterial filtration and breathability. Both masks passed all the tests made by Equilibrium. Native PDMS mask was certified as a level 2 mask for general use and recyclable PDMS mask was certified as a level 3 for general use.

4.4.1 Native PDMS mask

Table 11 presents the laboratory results that allowed the native PDMS mask to be certified. More additional information about the laboratory analysis and the conformability declaration can be found in appendix. The respective certification label is showed in Figure 24.

The result for bacterial filtration efficiency dictates the level of the mask. If the value is between 70 and 90%, the mask is classified as level 3 and if the value is higher than 90%, the mask is certified as level 2. Level 2 represents a higher level than level 3 in terms of protection.

Figure 24. *Certification label for the native PDMS mask.*

Table 11. *Results of the assays performed by Equilibrium for the native PDMS mask.*

Description	Methods	Results	Minimum requirements	Maximum allowable value
Visual Inspection	MI 179	Adequate	N/A	N/A
BFE (%)	EN 14683:2019+AC 2019-5.2.2	92.5±1.8%	≥70% (Level 3)	≥90% (Level 2)
Breathability (Pa/cm^2)	EN 14683:2019+AC 2019-5.2.3	21.8±8.7%	< 40 (Level 70%)	< 40 (Level 90%)
Cleaning	ISO 6330:2012	5 cleaning cycles, at 60 °C	N/A	N/A
Elastic resistance	MI 177	Resists	N/A	N/A

4.4.2 Recyclable PDMS mask

The results of the certification laboratory for the recyclable mask are described in Table 12. Additional information of the certification tests and procedures can be found in the appendix. The certification label of this mask can be observed in Figure 25.

From Table 12, the value measured of the BFE by the laboratory was 87.6%, which is lower than 90%, meaning that this mask was certified as level 3 mask for general use. It should be noted that this mask was not classified as level 2 only for 2.4%. Small changes in the geometry of the mask or in the used fabric may provide this mask the features needed to be certified as level 2.

Figure 25. *Certification label for the recyclable PDMS mask.*

Table 12. *Results of the assays performed by Equilibrium for the recyclable PDMS mask.*

Description	Methods	Results	Minimum requirements	Maximum allowable value
Visual Inspection	MI 179	Adequate	N/A	N/A
BFE (%)	EN 14683:2019+AC 2019-5.2.2	87.6±1.8%	≥70% (Level 3)	≥90% (Level 2)
Breathability (Pa/cm^2)	EN 14683:2019+AC 2019-5.2.3	<20 (QL)*±8.7%	< 40 (Level 70%)	< 40 (Level 90%)
Cleaning	ISO 6330:2012	5 cleaning cycles, at 60 °C	N/A	N/A
Elastic resistance	MI 177	Resists	N/A	N/A

*QL - Quantification limit

5. CONCLUSIONS AND FUTURE PERSPECTIVES

This chapter presents the conclusions drawn during the development of this dissertation work: a transparent face mask manufactured with combination of PDMS (including recyclable PDMS) and fabric. Moreover, future works to improve the developed mask are proposed.

5.1 Conclusion

This dissertation proposed the development of a transparent face mask made with PDMS. Based on the literature, it was concluded that developing a transparent respirator was expensive and implied complicated manufacture processes. Therefore, the development of a fabric mask with a transparent PDMS window was opted.

It is known that mixing PDMS with different ratios allowed to achieve different properties. Characterizing PDMS allowed to understand better those differences and compare properties between PDMS samples with different ratios. Based on the wettability assay, it was confirmed the hydrophobic nature of PDMS, and that the hydrophobicity decreases with increasing ratio. Additionally, transmittance tests allowed to prove the transparency of the material and no significant differences were observed between the different ratios. Finally, the mechanical tests allowed the measurement of Elastic Modulus of PDMS, and it was observed a decrease in this value with the increasing of the mixing ratio.

A method to recycle PDMS was developed. Using a manual food grater, it was possible to grate PDMS in such a way that it could be mixed with the liquid PDMS before curing. The characterization of the recycled PDMS allowed to conclude that recycled PDMS is slightly less hydrophobic, slightly less transparent, and more fragile than native PDMS, characteristics that accentuate with increasing the recycled PDMS percentage. Based on the characterization of native PDMS and recycled PDMS, it was chosen the 5:1 mixing ratio with 10% recycled PDMS to integrate the mask.

The fabric and the design of the mask was chosen taking in consideration the requirements of the standards. Two types of masks were fabricated. The first one was made with native PDMS of 5:1 mixing ratio and the second one was made with recycled PDMS of 5:1 mixing ratio and 10% of recycled PDMS solid particles. The masks were sent to a certification laboratory, and they were certified, respectively, as a level 2 and as level 3 mask

for general use. The masks are reusable as they can be cleaned in the washing machine at 60 °C, for 30 min, at least for five cycles.

It was developed a simple and cost-effective method to fabricate textile masks with a transparent PDMS window. The masks have proven to be effective in protecting against COVID-19, according to the certification output.

5.2 Future Perspectives

In a perspective of potentially launch the masks in the market, the cost per mask and the produced quantities per month were calculate. It was estimated that it could be possible to produce 8000 of native PDMS masks and 4000 recyclable PDMS masks for month. Additionally, each mask could be placed in the market for, respectively, 6 and 7 euros.

The masks herein developed present several limitations that can be overcome. For example, the mask has not anti-fogging properties. However, there are commercially available anti-fog spays, which the user can acquire and apply in the mask a couple of times a day. It is recommended that the spray is used both inside and outside of the PDMS surface. Like that it could be achieved the anti-fog properties and, at the same time, get an antibacterial treatment.

Despite the masks being certified for 5 wash cycles, the user can also choose for using a UV-C sterilizing equipment. The draw-back of using a UV-C is that the mask will only be sterilized and not cleaned. So, it is highly recommended that the user uses both types of sterilization in order to guarantee the maximum cleaning of the mask.

The next step can be the development of a 100% PDMS mask. The manufacturer process of Alenezi et al. [19] could be taken as an example. A manufacturer process proposal could be:

1. Numerical simulation in order to decide the more efficient design for the mask;
2. Draw the design in a CAD software and proceed to its 3D printing;
3. Create a mould with an epoxy resin;
4. Through silicone moulding, produce the PDMS mask.

An attempt to achieving a respirator relies in the use of filter caps must on the lateral or in the chin region, as acceptable by the design. The filter caps must be placed in such a way

that allows removing and replacing filters. This configuration should allow a good filtration efficiency and breathability. The inner surface of the respirator must be modified to be hydrophilic (for example, the method proposed by Gökaltun et al. [88] can be adopted), in order to acquire anti-fog properties. The outer surface must receive a hydrophobic treatment (like adding waxes [90]) so that the PDMS stays super hydrophobic and with antibacterial properties. Additionally, if the fogging persists, an integrated electronic system with a mini fan can be added to promote ventilation inside the mask. As for sterilization, UV-C is the recommended method. The respirator can be sold together with an appropriate UV sterilizing equipment so that when the user takes off the respirator, he can place it in the steriliser and proceed to its safe sterilization. Cleaning the respirator can be made with a moist cloth. In Figure 26, a representation of the described respirator can be observed. With the right resources, I believe that it is possible to develop such respirator.

Figure 26. *Schematic representation of a 100% PDMS mask.*

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APPENDIX

Declaration of certification of the native PDMS mask

EQUILIBRIUM
laboratório de controlo de qualidade e de processos

IPAC acreditação L0312 ISO/IEC 17025 Ensaios — ilac-MRA

Este boletim anula e substitui a versão anterior da data 22-03-2021

Boletim Analítico Nº:20214018 — Versão :2.0

Âmbito: Determinações em Amostras de Produtos Têxteis - MÁSCARAS

Boletim Definitivo

Requisitante: Otojal Estamparia Têxtil, Lda.
Morada: Rua da Cerquinha 202 - 4805-398 Guimarães, Portugal

Designação da Amostra: OTSMASK_SUSTENTAVEL_audiovisual

COLHEITA DE AMOSTRAS

Data:	10/03/2021	Colheita efectuada por:	Cliente
Hora de Colheita:	—	V/ Referência:	OTSMASK_SUSTENTAVEL_audiovisual
Método de Recolha:	—	Lote:	—
Tipo de Produto:	Máscara uso social	Tipologia:	Máscara reutilizável
Tamanho:	Adulto	Composição:	Tecido exterior INTERLOCK 100%pes rec, refª502085; 125g/m2; Entretela Interior 100%pes reciclado; refª 081; 58g/m2; Tecido interior VOILE 100% algodão orgânico; refª 102040; 75g/m2

ANÁLISE

Data de Entrada: 10/03/2021	Período de Análise: 10/03/2021 a 15/03/2021	Ref. Amostra: 20214018

Descrição	Métodos	Exp. Result.	Resultados/Incerteza	Lim. lei	VMR
Inspecção Visual	MI 176 *	—	Adequado	-	-
Bacterial filtration efficiency (BFE)	EN14683:2019+AC 2019-5.2.2	%	92.5 ±1,8%	≥70% (Nível70%)	≥90%(Nível90%)
Breathability (Differencial pressure)	EN14683:2019+AC 2019-5.2.3	Pa/cm2	21,8 ±8,7% (U ensaio)	Nível 70% <40	Nível 90% <40
Lavagem e Secagem Doméstica	ISO 6330:2012 *	—	Ensaios após 5 (cinco) ciclos a 60ºC	—	—
Resistência do Elástico	MI 177 *	-	Resiste	-	-

APRECIAÇÃO

Leia-se os valores LimLei e VMR, os valores definidos na Cf INfo n.009/2020, de 13/04/2020 da DGS, relativa "COVID-19:fase mitigação-uso másc. na comunidade" e CWA 17553:2020.Métodos analíticos pela EN 14683:2019+AC2019.

Após comparação, dos resultados obtidos nos ensaios realizados com os limites tabelados, a amostra analisada encontra-se CONFORME.

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Designação da Amostra: OTSMASK_SUSTENTAVEL_audiovisual

COLHEITA DE AMOSTRAS

Data:	10/03/2021	Colheita efectuada por:	Cliente
Hora de Colheita:	---	V/ Referência:	OTSMASK_SUSTENTAVEL_audiovisual
Método de Recolha:	---	Lote:	---
Tipo de Produto:	Máscara uso social	Tipologia:	Máscara reutilizável
Tamanho:	Adulto	Composição:	Tecido exterior INTERLOCK 100%pes rec, refª502085; 125g/m2; Entretela Interior 100%pes reciclado; refª 081; 58g/m2; Tecido interior VOILE 100% algodão orgânico; refª 102040; 75g/m2

ANÁLISE

Data de Entrada: 10/03/2021	Período de Análise: 10/03/2021 a 15/03/2021	Ref. Amostra: 20214018

Descrição	Métodos	Exp. Result.	Resultados/Incerteza	Lim. lei	VMR

OBSERVAÇÕES

BACTERIAL FILTRATION EFFICIENCY (Filtration) _EN14683:2019+AC 2019:
Condições teste: Temperatura e Humidade relativa // Test conditions: Temperature and Relative humidity: 21±5ºC / 85± 5 %
Área de cada réplica/ Dimensions of the test specimens: 49cm2 (5 réplicas/ test specimens)
Superfície da amostra testada/ Side of the test specimen facing the challenge aerossol: interna / intern
Air flow rate: 28.3 l/min.
MPS 2.9
Valor médio da contagem total de 2 controles positivos/Average of the total quantification of 2 positive controls (CFU): 2745
Valor médio da contagem do controle negativo/Average of the quantification of the negative control (CFU): 5
Réplica 1, Réplica 2, Réplica 3, Réplica 4, Réplica 5 (%) // Test specimen 1 ,Test specimen 2,Test specimen 3,Test specimen 4,Test specimen 5 (%)
91.0
91.2
95.2
95.3
91.7

DIFFERENCIAL PRESSURE (Breathability) _EN14683:2019+AC 2019:
Condições teste: Temperatura e Humidade relativa // Test conditions: Temperature and Relative humidity: 21±5ºC / 85± 5 %
Área de cada réplica// Dimensions of the test specimens: 4.9cm2 (5 réplicas/ 5 test specimens)
Número de áreas por réplica// Number of áreas per specimen: 5 áreas por réplica (A,B,C,D,E)// 5 áreas for test specimen (A,B,C,D,E)
Superfície da amostra testada: Teste realizado com a direcção do fluxo do interior para o exterior. Localização lateral e central. // Number and general location of the areas of the mask the differential measurements were taken: Test performed with the direction of flow from the inside to the outside. Side and central location.
Air flow rate: 8L/min
Réplica 1 (área A,B,C,D,E), Réplica 2 (área A,B,C,D,E), Réplica 3 (área A,B,C,D,E), Réplica 4 (área A,B,C,D,E) Réplica 5 (área A,B,C,D,E) //Test specimen 1 (área A,B,C,D,E), Test specimen 2 (área A,B,C,D,E),Test specimen 3 (área A,B,C,D,E), Test specimen 4 (área A,B,C,D,E), Test specimen 5 (área A,B,C,D,E) Unidades (Pa/cm2)
21,4
21,4
22,4
22,4
21,4

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Boletim Analítico Nº:20214018

Versão :2.0

Âmbito: Determinações em Amostras de Produtos Têxteis - MÁSCARAS

Boletim Definitivo

Requisitante: Otojal Estamparia Têxtil, Lda.
Morada: Rua da Cerquinha 202 - 4805-398 Guimarães, Portugal

Designação da Amostra: OTSMASK_SUSTENTAVEL_audiovisual

COLHEITA DE AMOSTRAS

Data:	10/03/2021	Colheita efectuada por:	Cliente
Hora de Colheita:	---	V/ Referência:	OTSMASK_SUSTENTAVEL_audiovisual
Método de Recolha:	---	Lote:	---
Tipo de Produto:	Máscara uso social	Tipologia:	Máscara reutilizável
Tamanho:	Adulto	Composição:	Tecido exterior INTERLOCK 100%pes rec, refª502085; 125g/m2; Entretela Interior 100%pes reciclado; refª 081; 58g/m2; Tecido interior VOILE 100% algodão orgânico; refª 102040; 75g/m2

ANÁLISE

Data de Entrada: 10/03/2021	Período de Análise: 10/03/2021 a 15/03/2021	Ref. Amostra: 20214018

Descrição	Métodos	Exp. Result.	Resultados/Incerteza	Lim. lei	VMR

CICLOS LAVAGEM E SECAGEM DOMÉSTICA/ Domestic washing and drying :
Tipo máquina: eixo horizontal, carga frontal / Type machine: horizontal axis, front-loading
Balastro: carga têxtil 100% algodão / Ballast used: 100% cotton
Detergente: Det. Ref. 3 / Detergent: Det. Ref. 3
Massa de carga seca total: 2.0 ±0.1 Kg / Total dry mass: 2.0 ±0.1 Kg
Secagem por suspensão / Drying procedure: Line dry

RESISTÊNCIA AO ELÁSTICO/ Head harness strength :
Verificação da Resistência a 5 ciclos de colocação e remoção, em 3 sujeitos de ensaio, com diferentes morfologias.
// Verification of resistance to 5 cycles of placement and removal, in 3 test subjects, with different morphologies.

Inspecão Visual/ Visual Inspection:
- Ajuste do clipe nasal // Nasal clip adjustment
- Rasgos // Rips,
- Deformações // Deformations,
- Desgaste // Wear,
- Cobertura Facial // Facial Coverage

Nota V2: Alteração do cliente descrito no boletim, a pedido do cliente.

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Boletim Analítico Nº:20214018

Versão :2.0

Âmbito: Determinações em Amostras de Produtos Têxteis - MÁSCARAS

Boletim Definitivo

Requisitante: Otojal Estamparia Têxtil, Lda.
Morada: Rua da Cerquinha 202 - 4805-398 Guimarães, Portugal

Designação da Amostra: OTSMASK_SUSTENTAVEL_audiovisual

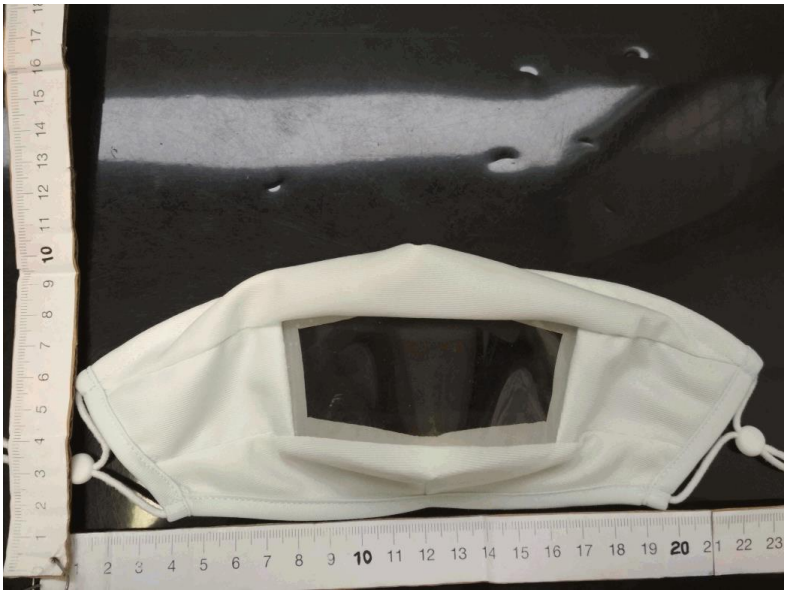
COLHEITA DE AMOSTRAS

Data:	10/03/2021	Colheita efectuada por:	Cliente
Hora de Colheita:	--	V/ Referência:	OTSMASK_SUSTENTAVEL_audiovisual
Método de Recolha:	--	Lote:	--
Tipo de Produto:	Máscara uso social	Tipologia:	Máscara reutilizável
Tamanho:	Adulto	Composição:	Tecido exterior INTERLOCK 100%pes rec, refª502085; 125g/m2; Entretela Interior 100%pes reciclado; refª 081; 58g/m2; Tecido interior VOILE 100% algodão orgânico; refª 102040; 75g/m2

ANÁLISE

Data de Entrada: 10/03/2021	Período de Análise: 10/03/2021 a 15/03/2021	Ref. Amostra: 20214018

Descrição	Métodos	Exp. Result.	Resultados/Incerteza	Lim. lei	VMR



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EQUILIBRIUM
laboratório de controlo de qualidade e de processos

IPAC acreditação L0312 ISO/IEC 17025 Ensaios

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Este boletim anula e substitui a versão anterior da data 22-03-2021

Boletim Analítico Nº:20214018

Versão :2.0

Âmbito: Determinações em Amostras de Produtos Têxteis - MÁSCARAS

Boletim Definitivo

Requisitante: Otojal Estamparia Têxtil, Lda.
Morada: Rua da Cerquinha 202 - 4805-398 Guimarães, Portugal

Designação da Amostra: OTSMASK_SUSTENTAVEL_audiovisual

COLHEITA DE AMOSTRAS

Data:	10/03/2021	Colheita efectuada por:	Cliente
Hora de Colheita:	---	V/ Referência:	OTSMASK_SUSTENTAVEL_audiovisual
Método de Recolha:	---	Lote:	---
Tipo de Produto:	Máscara uso social	Tipologia:	Máscara reutilizável
Tamanho:	Adulto	Composição:	Tecido exterior INTERLOCK 100%pes rec, refª502085; 125g/m2; Entretela Interior 100%pes reciclado; refª 081; 58g/m2; Tecido interior VOILE 100% algodão orgânico; refª 102040; 75g/m2

ANÁLISE

Data de Entrada: 10/03/2021	Período de Análise: 10/03/2021 a 15/03/2021	Ref. Amostra: 20214018

Descrição	Métodos	Exp. Result.	Resultados/Incerteza	Lim. lei	VMR

O ensaio assinalado com (*) não se encontra no âmbito da acreditação do laboratório.

A amostragem efectuada não se encontra incluída no âmbito da acreditação.

NP: Norma Portuguesa; SMEWW: Standart Methods for the Examination of Water and Wastewater; ISO: Internacional Standard Organization; AFNOR: Association Francaise de Normalisation; LAE: L'Analyse des Eaux, Rodier 8e Édition; EN: Norma Europeia; DIN: Deutshes Institut fur Normung; EPA: Environmental Protection Agency; ASTM: American Society for Testing and Materials; MI: Método Interno; N/A: Não aplicável; LQ: Limite de Quantificação; LD: Limite de Deteção; UFC: Unidades formadoras de colónias; VMR: Valor máximo recomendado; VMA: Valor máximo admissível; VP: Valor Paramétrico; Nos resultados obtidos por cálculo com base em resultados individuais, serão contabilizadas as parcelas quantificáveis desprezando as parcelas <LQ. Se todas as parcelas forem <LQ, o valor emitido será o LQ do método. O resultado pode ser apresentado com Medida de Incerteza Expandida, do Ensaio (não contemplando a da amostragem), a um nível de confiança de 95%, k=2, quando esta aparece associada ao resultado.O laboratório não contabiliza a incerteza no método na declaração de conformidade. A apresentação do resultado como ≤ ao LQ corresponde à faixa de guarda associada à incerteza do método. Os dados fornecidos pelo cliente são da sua responsabilidade (descritos no campo: "Colheitas" e "Designação Amostras") .Os resultados aplicam-se à amostra conforme recepcionada/Results apply to the sample as received.

EMISSÃO

Matosinhos, 14 de abril de 2021

O Director do Laboratório

Maria Cristina Antão, Dra.

(Este Boletim Analítico foi assinado digitalmente)

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DECLARATION

Sample features under COVID-19

OTOJAL- ESTAMPARIA TÊXTIL, LDA

Rua da Cerquinha, 202

4805-398 Ronfe, Portugal

OTSMASK_SUSTAINABLE_audiovisual

LEVEL 90% | 5 WASH CYCLES 60ºC

TEST REPORT: Nº 20214018 of 14/04/2021

It is stated for the due effects that the company identified above performs as analytical determinations within the scope of COVID-19, under the document published by CEN Workshop Agreement CWA 17553:2020 "Community face coverings - Guide to minimum requirements, methods of testing and use", at the EQUILIBRIUM Laboratory.

The results of the Bacterial Filtration Efficiency and Breathability tests are within the analytical requirements under CWA17553:2020 for level 90% , 5 wash cycles 60ºC

For the purposes of laboratory analysis, evaluation and issuing of the appraisal with the symbol COVID-19 CWA17553:2020, the identified company declared that the sample sent to the laboratory, with the denomination of sale / reference / batch production analyzed corresponds to the characteristics of the product to be placed on the market.

It is up to the company to be responsible for the veracity / correspondence of the sample to be sold with the sample sent to EQUILIBRIUM Laboratory, being responsible for maintaining the characteristics analyzed in subsequent productions. In case of changes you are not allowed to use the symbol COVID-19 CWA 17553:2020 EQUILIBRIUM.

This textile article is intended to serve as a complementary protection to measures of social distance within the scope of the control of COVID-19. It connot be used by health professionals in the exercise of their functions, it is not a medical device or PPE (the surgical masks Reg. EU/2017/745 or Reg. EPI EU/2016/425 do not comply).
It is safeguarded specific changes to the CWA17553:2020 of each country.

Matosinhos, 14 of April 2021

Laboratory Director, Msc Cristina Antão

E Q U I L I B R I U M
laboratório de controlo de qualidade e de processos

EQUILIBRIUM – Laboratório de Controlo de Qualidade e de Processos, Lda. Praceta João Villaret, 183 4460-37 Senhora da Hora – PORTUGAL F.R.7.5.250-2

Declaration of certification of the recyclable PDMS mask

EQUILIBRIUM
laboratório de controlo de qualidade e de processos

IPAC acreditação L0312 ISO/IEC 17025 Ensaios

ilac-MRA

Boletim Analítico Nº:20218332

Versão :1.0

Âmbito: Determinações em Amostras de Produtos Têxteis - MÁSCARAS

Boletim Definitivo

Requisitante: Otojal Estamparia Têxtil, Lda.
Morada: Rua da Cerquinha 202 - 4805-398 Guimarães, Portugal

Designação da Amostra: OTSMASK_SUSTENTAVEL_audiovisual

COLHEITA DE AMOSTRAS

Data:	20/05/2021	Colheita efectuada por:	Cliente
Hora de Colheita:	---	V/ Referência:	OTSMASK_SUSTENTAVEL_audiovisual
Método de Recolha:	---	Lote:	---
Tipo de Produto:	Máscara uso social	Tipologia:	Máscara reutilizável
Tamanho:	Adulto	Composição:	Tecido exterior INTERLOCK 100%pes rec, refª502085; 125g/m2; Interior 100%pes reciclado; refª 081; 58g/m2; Tecido interior VOILE 100% algodão orgânico; refª 102040; 75g/m2

ANÁLISE

Data de Entrada: 20/05/2021	Período de Análise: 20/05/2021 a 25/05/2021	Ref. Amostra: 20218332

Descrição	Métodos	Exp. Result.	Resultados/Incerteza	Lim. lei	VMR
Inspecção Visual	MI 176 *	—	Adequado	-	-
Bacterial filtration efficiency (BFE)	EN14683:2019+AC 2019-5.2.2	%	87,6 ±1.8%	≥70% (Nivel70%)	≥90%(Nivel90%)
Breathability (Differencial pressure)	EN14683:2019+AC 2019-5.2.3	Pa/cm2	<20 (LQ) ±8.7%	<40 (Nivel 70%)	<40 (Nivel 90%)
Lavagem e Secagem Doméstica	ISO 6330:2012 *	—	Ensaios após 5 (cinco) ciclos a 60ºC	---	—
Resistência do Elástico	MI 177 *	—	Resiste	-	-

APRECIAÇÃO

Leia-se os valores LimLei e VMR, os valores definidos na Cf INfo n.009/2020, de 13/04/2020 da DGS, relativa "COVID-19:fase mitigação-uso másc. na comunidade" e CWA 17553:2020.Métodos analíticos pela EN 14683:2019+AC2019.

Após comparação, dos resultados obtidos nos ensaios realizados com os limites tabelados, a amostra analisada encontra-se CONFORME.
//

Read the values LimLei and VMR, the values defined in CWA 17553:2020, relative to "Community face coverings - Guide to minimum requirements, methods of testing and use". Analytical methods on EN 14683: 2019.

After comparing the results obtained in the tests performed with the tabulated limits, the sample analyzed is QUALIFIED.

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Boletim Analítico Nº:20218332

Versão :1.0

Âmbito: Determinações em Amostras de Produtos Têxteis - MÁSCARAS

Boletim Definitivo

Requisitante: Otojal Estamparia Têxtil, Lda.
Morada: Rua da Cerquinha 202 - 4805-398 Guimarães, Portugal

Designação da Amostra: OTSMASK_SUSTENTAVEL_audiovisual

COLHEITA DE AMOSTRAS

Data:	20/05/2021	Colheita efectuada por:	Cliente
Hora de Colheita:	---	V/ Referência:	OTSMASK_SUSTENTAVEL_audiovisual
Método de Recolha:	---	Lote:	---
Tipo de Produto:	Máscara uso social	Tipologia:	Máscara reutilizável
Tamanho:	Adulto	Composição:	Tecido exterior INTERLOCK 100%pes rec, refª502085; 125g/m2; Interior 100%pes reciclado; refª 081; 58g/m2; Tecido interior VOILE 100% algodão orgânico; refª 102040; 75g/m2

ANÁLISE

Data de Entrada: 20/05/2021	Período de Análise: 20/05/2021 a 25/05/2021	Ref. Amostra: 20218332

Descrição	Métodos	Exp. Result.	Resultados/Incerteza	Lim. lei	VMR

OBSERVAÇÕES

BACTERIAL FILTRATION EFFICIENCY (Filtration) _EN14683:2019+AC 2019:
Condições teste: Temperatura e Humidade relativa // Test conditions: Temperature and Relative humidity: 21±5ºC / 85± 5 %
Área de cada réplica/ Dimensions of the test specimens: 49cm2 (5 réplicas/ test specimens)
Superfície da amostra testada/ Side of the test specimen facing the challenge aerossol: interna / intern
Air flow rate: 28.3 l/min.
MPS 2.9
Valor médio da contagem total de 2 controles positivos/Average of the total quantification of 2 positive controls (CFU): 2258
Valor médio da contagem do controle negativo/Average of the quantification of the negative control (CFU): 2
Réplica 1, Réplica 2, Réplica 3, Réplica 4, Réplica 5 (%) // Test specimen 1 ,Test specimen 2,Test specimen 3,Test specimen 4,Test specimen 5 (%)
88.0
88.3
87.5
86.5
87.5

DIFFERENCIAL PRESSURE (Breathability) _EN14683:2019+AC 2019:
Condições teste: Temperatura e Humidade relativa // Test conditions: Temperature and Relative humidity: 21±5ºC / 85± 5 %
Área de cada réplica// Dimensions of the test specimens: 4.9cm2 (5 réplicas/ 5 test specimens)
Número de áreas por réplica// Number of áreas per specimen: 5 áreas por réplica (A,B,C,D,E)// 5 áreas for test specimen (A,B,C,D,E)
Superfície da amostra testada: Teste realizado com a direcção do fluxo do interior para o exterior. Localização lateral e central. // Number and general location of the areas of the mask the differential measurements were taken: Test performed with the direction of flow from the inside to the outside. Side and central location.
Air flow rate: 8L/min
Réplica 1 (área A,B,C,D,E), Réplica 2 (área A,B,C,D,E), Réplica 3 (área A,B,C,D,E), Réplica 4 (área A,B,C,D,E) Réplica 5 (área A,B,C,D,E)
//Test specimen 1 (área A,B,C,D,E), Test specimen 2 (área A,B,C,D,E),Test specimen 3 (área A,B,C,D,E), Test specimen 4 (área A,B,C,D,E), Test specimen 5 (área A,B,C,D,E) Unidades (Pa/cm2)
16,3
17,3
15,3
18,4
18,4

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CICLOS LAVAGEM E SECAGEM DOMÉSTICA/ Domestic washing and drying :
Tipo máquina: eixo horizontal, carga frontal / Type machine: horizontal axis, front-loading
Balastro: carga têxtil 100% algodão / Ballast used: 100% cotton
Detergente: Det. Ref. 3 / Detergent: Det. Ref. 3
Massa de carga seca total: 2.0 ±0.1 Kg / Total dry mass: 2.0 ±0.1 Kg
Secagem por suspensão / Drying procedure: Line dry

RESISTÊNCIA AO ELÁSTICO/ Head harness strength :
Verificação da Resistência a 5 ciclos de colocação e remoção, em 3 sujeitos de ensaio, com diferentes morfologias.
// Verification of resistance to 5 cycles of placement and removal, in 3 test subjects, with different morphologies.

Inspecão Visual/ Visual Inspection:
- Ajuste do clipe nasal // Nasal clip adjustment
- Rasgos // Rips,
- Deformações // Deformations,
- Desgaste // Wear,
- Cobertura Facial // Facial Coverage

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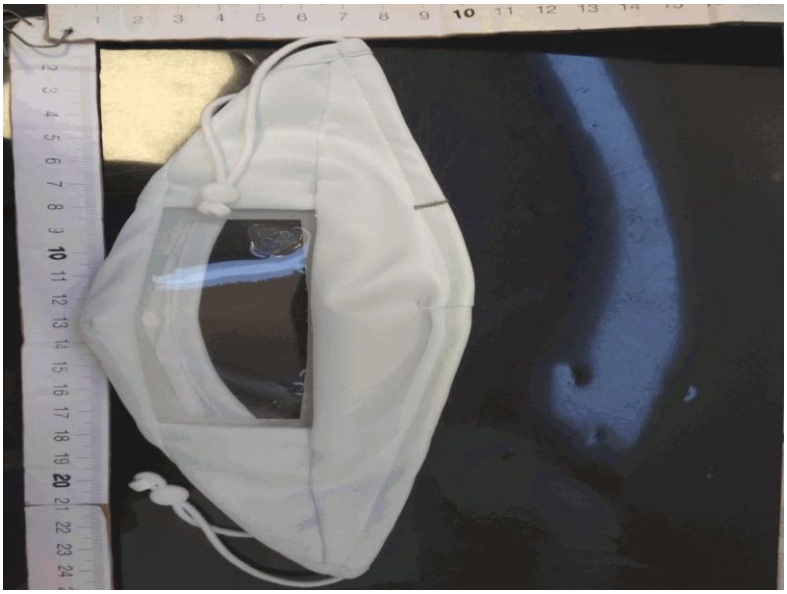
Descrição	Métodos	Exp. Result.	Resultados/Incerteza	Lim. lei	VMR



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O ensaio assinalado com (*) não se encontra no âmbito da acreditação do laboratório.

A amostragem efectuada não se encontra incluída no âmbito da acreditação.

NP: Norma Portuguesa; SMEWW: Standart Methods for the Examination of Water and Wastewater; ISO: Internacional Standard Organization; AFNOR: Association Francaise de Normalisation; LAE: L'Analyse des Eaux, Rodier 8e Édition; EN: Norma Europeia; DIN: Deutshes Institut fur Normung; EPA: Environmental Protection Agency; ASTM: American Society for Testing and Materials; MI: Método Interno; N/A: Não aplicável; LQ: Limite de Quantificação; LD: Limite de Deteção; UFC: Unidades formadoras de colónias; VMR: Valor máximo recomendado; VMA: Valor máximo admissível; VP: Valor Paramétrico; Nos resultados obtidos por cálculo com base em resultados individuais, serão contabilizadas as parcelas quantificáveis desprezando as parcelas <LQ. Se todas as parcelas forem <LQ, o valor emitido será o LQ do método. O resultado pode ser apresentado com Medida de Incerteza Expandida, do Ensaio (não contemplando a da amostragem), a um nível de confiança de 95%, k=2, quando esta aparece associada ao resultado.O laboratório não contabiliza a incerteza no método na declaração de conformidade. A apresentação do resultado como ≤ ao LQ corresponde à faixa de guarda associada à incerteza do método. Os dados fornecidos pelo cliente são da sua responsabilidade (descritos no campo: "Colheitas" e "Designação Amostras") .Os resultados aplicam-se à amostra conforme recepcionada/Results apply to the sample as received.

EMISSÃO

Matosinhos, 25 de maio de 2021

O Director do Laboratório

Maria Cristina Antão, Dra.

(Este Boletim Analítico foi assinado digitalmente)

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DECLARATION

Sample features under COVID-19

OTOJAL ESTAMPARIA TÊXTIL, LDA

Rua da Cerquinha, 202

4805-398 Guimarães, Portugal

OTSMASK_SUSTAINABLE_audiovisual

LEVEL 70% | 5 WASH CYCLES 60ºC

TEST REPORT: Nº 20218332 of 25/05/2021

It is stated for the due effects that the company identified above performs as analytical determinations within the scope of COVID-19, under the document published by CEN Workshop Agreement CWA 17553:2020 "Community face coverings - Guide to minimum requirements, methods of testing and use", at the EQUILIBRIUM Laboratory.

The results of the Bacterial Filtration Efficiency and Breathability tests are within the analytical requirements under CWA17553:2020 for level 70% , 5 wash cycles 60ºC

For the purposes of laboratory analysis, evaluation and issuing of the appraisal with the symbol COVID-19 CWA17553:2020, the identified company declared that the sample sent to the laboratory, with the denomination of sale / reference / batch production analyzed corresponds to the characteristics of the product to be placed on the market.

It is up to the company to be responsible for the veracity / correspondence of the sample to be sold with the sample sent to EQUILIBRIUM Laboratory, being responsible for maintaining the characteristics analyzed in subsequent productions. In case of changes you are not allowed to use the symbol COVID-19 CWA 17553:2020 EQUILIBRIUM.

This textile article is intended to serve as a complementary protection to measures of social distance within the scope of the control of COVID-19. It connot be used by health professionals in the exercise of their functions, it is not a medical device or PPE (the surgical masks Reg. EU/2017/745 or Reg. EPI EU/2016/425 do not comply).
It is safeguarded specific changes to the CWA17553:2020 of each country.

Matosinhos, 26 of May 2021

Laboratory Director, Msc Cristina Antão

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