

# **Properties characterization of PDMS/Beeswax composite**

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Dissertation presented to Escola Superior de Tecnologia e Gestão of Instituto Politécnico de Bragança to obtain the Master Degree in Industrial Engineering

Supervised by:

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**Bragança**

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## Resumo

O polidimetilsiloxano (PDMS) é um dos elastómeros pertencente aos polímeros que mais tem recebido atenção, por ser um material com boa estabilidade térmica, biocompatibilidade, flexibilidade, baixo custo e características hiperplásticas. Assim como o PDMS, a cera de abelha, também, tem atraído a atenção dos investigadores, por se tratar de um material biodegradável, termicamente estável e de origem natural. Esses materiais podem ser utilizados em áreas como sistemas microfluídicos, dispositivos médicos, componentes eletrônicos, entre outros. O PDMS misturado com cera de abelha, mostra-se capaz de melhorar as propriedades hidrofóbicas, resistência à abrasão e corrosão, estabilidade térmica e transparência a alta temperatura. Porém, o processo de fabricação utilizado para misturar PDMS e ceras requer algumas etapas, como aquecer, misturar e desgaseificar, contudo, os métodos convencionais não seguem um processo normalizado, originando produtos com baixa repetibilidade. Para contornar esta limitação, desenvolveu-se e construiu-se uma câmara de vácuo com o objetivo de otimizar o processo de fabricação. Outro fator importante é a utilização da cera de abelha por ser um produto natural, a composição é diferente dependendo do clima e da região. Por esse motivo, neste estudo foi realizada a caracterização química da cera de abelha. Posteriormente, foram realizados testes experimentais com o compósito de PDMS e cera de abelha. O fabrico das amostras foi efetuado utilizando a câmara de vácuo multifuncional desenvolvida nesta dissertação. As amostras foram submetidas a ensaios de tração, dureza, DMA, TGA, espectrometria e de molhabilidade com o intuito de analisar as propriedades mecânicas, óticas e de molhabilidade. A fabricação da câmara de vácuo multifuncional permitiu a produção das amostras com propriedades mais uniformes e tornou o processo mais eficiente. No ensaio de DMA, o compósito mostrou uma estabilidade térmica até os 200°C, juntamente com a alta transparência a 80°C, quando comparado ao PDMS puro. No ensaio de molhabilidade, o compósito provou aumentar o ângulo de contacto perto dos 150°C, apresentando uma superfície super-hidrofóbica.

## **Abstract**

Polydimethylsiloxane (PDMS) is one of the elastomers belonging to the polymers that has received the most attention, as it is a material with good thermal stability, biocompatibility, flexibility, low cost and hyperplastic characteristics. As well as PDMS, beeswax, too, has attracted the attention of researchers, as it is a biodegradable material, thermally stable and of natural origin. These materials can be used in areas such as microfluidic systems, medical devices, electronic components, among others. PDMS mixed with beeswax is able to improve hydrophobic properties, abrasion and corrosion resistance, thermal stability and high temperature transparency. However, the manufacturing process used to mix PDMS and waxes requires some steps, such as heating, mixing and degassing, however, conventional methods do not follow a standardized process, resulting in products with low repeatability. To overcome this limitation, a vacuum chamber was developed and built with the objective of optimizing the manufacturing process. Another important factor is the use of beeswax, as it is a natural product, the composition is different depending on the climate and region. For this reason, in this study, the chemical characterization of beeswax was performed. Subsequently, experimental tests were carried out with the composite of PDMS and beeswax. Samples were manufactured using the multifunctional vacuum chamber developed in this dissertation. The samples were submitted to tensile, hardness, DMA, TGA, spectrometry and wettability tests in order to analyze the mechanical, optical and wettability properties. The manufacture of the multifunctional vacuum chamber allowed the production of samples with more uniform properties and made the process more efficient. In the DMA test, the composite showed thermal stability up to 200°C, together with high transparency at 80°C, when compared to pure PDMS. In the wettability test, the composite proved to increase the contact angle close to 150°, presenting a super-hydrophobic surface.

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## List of Abbreviations and Symbols

WCA	Contact water angle
SA	Slip angle
TS	Tensile strength
UTS	Ultimate tensile strength
MVC	Multifunctional vacuum chamber

# Chapter 1

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## 1.1 Introduction

Polymeric materials have been used since ancient times, but at that time only natural polymeric materials were used. Polymers are macromolecules formed from smaller structural units called monomers. Monomers are low molecular mass molecules that from polymerization reactions generate the polymeric macromolecule. It was only in the 20th century that the artificial polymerization processes took shape, since then the processes have undergone improvement to obtain plastics, rubbers and resins thanks to an increasingly complex molecular engineering being widely used as a basis in various industrial products [1]. Furthermore, the polymer industry has grown rapidly and is now larger than aluminum, copper and some other major materials processing industries. Among them, an important subclass of materials is elastomers [2]. They are weakly cross-linked polymers, usually with low Young's modulus, supporting high yield deformation and being able to restore the original shape when a stress, which caused the deformation, is present. Examples include natural and synthetic rubbers, silicone elastomers and other copolymers [3]. The elastomers have attracted the attention of scientists with polydimethylsiloxane (PDMS), which is a mixture of linear siloxane polymers that are fully methylated, consisting of units of the formula  $\text{H}(\text{CH}_3)_2\text{SiO}$ , stabilized by terminal units of the formula  $(\text{CH}_3)_2\text{SiO}$ . This component belongs to a group of polymeric organosilicones called silicones. PDMS is the most used silicone based on organic polymers, it presents a clear, non-flammable, non-toxic compound [4].

Biodegradable materials are another material that has been receiving attention due to their short decomposition time. Beeswax has been standing out among materials of natural origin due to its excellent chemical and thermal properties [5]. Beeswax is synthesized from honey sugars and secreted in liquid form by glands located in the abdomen, when it comes into contact with the atmosphere it solidifies instantly [6]. Like PDMS, beeswax has some similar characteristics of non-toxic and biodegradable [7].

A wide range of applications is concentrated by the desirable properties presented by this polymer such as: High flexibility, biocompatibility, chemically and thermally stable, optically transparent, corrosion resistance, repeatability, abrasion resistance, gas

permeability and low cost [8]. Thus, its wide application focuses on contact lenses, cosmetics, food and lubricants, electronic devices, microfluidic devices, filtration membranes, sensors, blood analogues, sealing agents, biomodels, optical and thermal devices, coatings, organ platforms on a chip and biomadical devices. Its main characteristics of surface coatings are to increase hydrophobicity, water repellent, anti-ice, anti-fog, anti-fouling and self-cleaning [9]. However, the manufacturing process is relatively simple, or even complex depending on the mixture, but there is no improvement in this conventional method used since then.

There is a surprising growth rate in studies involving beeswax in fact that its application is in several areas, such as in the food, cosmetic, pharmaceutical and medicinal industries [6], [10]. Among these areas, the main applications are in packaging for food preservation [7], medicines and waterproofing [11]. These applications are justified by the fact that beeswax retains excellent properties, such as biocompatible [12], extremely ductile, antibacterial and humidity resistance [13].

PDMS has been widely used in super-hydrophobic coatings, consisting of surfaces with low surface energy. Li et al proposed a pervaporation membrane prepared by coating SiO<sub>2</sub>/PDMS on the hollow surface of the polyvinylidene fluoride membrane. The membranes showed improved mechanical properties and good phenol permeation under water, as the contact angle with water increased by almost 50% [14]. Pakzad et al presented a modified silicon nanoparticle surface with PDMS and beeswax. Both surfaces have improved hydrophobic property, corrosion resistance and durability in acidic solutions. Having potential application in liquid separation membranes, waterproof and self-cleaning surface coatings [9].

Another field of application, due to its transparent characteristics, PDMS has been applied in optical components, for example, in replacement of natural human lenses, having the possibility of reaching a refractive index close to the natural human lens 1.433. Elastomer can prevent a secondary loss of vision caused by migration of lens epithelial cells during surgery. Riehle et al reported an index of refraction of 1,403 for unmodified PDMS and 1,434 for PDMS and polysiloxane-urea [15]. Cruz-Felix et al reported a refractive index around 1,408 to 1,445 for PDMS with different healing processes. Other optical applications are in microfluidics, photovoltaic panels due to excellent thermal stability with a high degree of transparency [16]. Syafiq et al proposed a hybrid coating

with PDMS and APTES by a simple immersion process resulting in a hydrophobic surface with a contact angle with water above  $100^\circ$  and transmittance above 90% in the visible region. The results were satisfactory and showed excellent transparency properties and self-cleaning, anti-fog properties in indoor and outdoor environments [17]. Park et al also manufactured a laminated film, produced with graphene, paraffin and PDMS with excellent transparency properties in the visible region of the light spectrum. With excellent results, these coatings have enormous promising potential in applications for photovoltaic panels. However, these processes involve chemical modifications during the fabrication of films/coatings that may not be feasible to use [18].

Biodegradable products have been studied and tested to replace petroleum-derived products with the purpose of preserving nature, so beeswax has been tested and analyzed for uses in various sectors. Biodegradable products have been studied and tested to replace petroleum-derived products with the purpose of preserving nature, which is why beeswax has been tested and analyzed for uses in various sectors. Oliveira et. al. they produced coatings to maintain the chemical properties of fruits and vegetables. The biofilm containing 10% of bee certain presented better results in the water vapor transmission rate, 80% in the elasticity and decreased 15% in the solubility, ensuring adequate fruit ripening for 15 days [19]. Reis et. al. they proposed biodegradable tray coatings by immersion in beeswax emulsion. Trays with 1% in beeswax resulted in higher tensile strength, lower water solubilization capacity and reduced permeability of grottos and fresh vegetables [7].

## **1.2 Motivation and Goals**

Despite technological advances in processing and improving polymers in general, the manufacturing method for polydimethylsiloxane (PDMS) has not followed the same trend. Using PDMS in favor of the desired characteristics of biocompatibility, thermally stable, high flexibility and transparent, these properties are extremely important for some applications, but this silicone does not have very strong mechanical properties and has the potential to increase its applicability several times by combining with other materials, for example, fibers, additives, waxes, blends, micro and nanoparticles. However, the manufacturing process must be adaptable to each combination due to the chemical composition of each element, for example, the manufacturing process of PDMS and waxes requires heating because waxes in general at room temperature are in the solid state

of matter, making interaction between them difficult. In order to manufacture this composite of PDMS and beeswax its processing requires 3 main steps, such as:

1. Manual heating and mixing of PDMS and beeswax.
2. Add curing agent and hand-mix again.
3. Pour into molds and degas.

The conventional manufacturing method has no record of standardization, or even control of these parameters, with manual mixing, heating in a water bath without temperature control and degassing in a single step.

Therefore, the overall objective of this work is to develop a multifunctional vacuum chamber to improve manufacturing efficiency, followed by manufacturing and characterization of the PDMS/beeswax composite using the multifunctional vacuum chamber. This general objective is subdivided into the following specific objectives:

1. Development, design and manufacture a multifunctional vacuum chamber.
2. Manufacture of PDMS and beeswax composite specimens.
3. Mechanical, optical and wettability characterization of the manufactured composite.

### **1.3 Structure**

This dissertation is divided into six chapters. In the first chapter, the base materials that will be studied in this work were presented. In this step, the characteristics that polydimethylsiloxane has in common with beeswax are provided, followed by individual information, its properties and daily applications are also presented. At this stage, the motivation for carrying out the work and the proposed objectives to be achieved are also reported.

The second chapter covers a general review of polydimethylsiloxane (PDMS) with its main characteristics and properties coming from the mixture of other elements. The focus of the review is to demonstrate the polymer's ability to mix with other materials to obtain composites with excellent chemical and thermal properties, also for being a biocompatible and transparent material. This chapter is divided into several areas that PDMS has combination with other materials, for example waxes, particles, fibers,

additives and blends, presenting the results obtained and the wide range of applications that PDMS is inserted.

The third chapter presents the development and construction of the multifunctional vacuum chamber to improve the PDMS manufacturing process with other materials that need parameter control, for example, temperature control, mixing speed control and internal pressure control. The focus of this multifunctional vacuum chamber is to minimize the manufacturing process making some steps faster and simultaneously making the overall process more efficient. In this step, the comparison of manufacturing by the conventional method and the method using the multifunctional vacuum chamber is also carried out.

The fourth chapter presents the chemical characterization of the beeswax used to manufacture the composite of PDMS with beeswax, as the wax is supplied by a local beekeeper (Bragança, Portugal).

The fifth chapter presents the fabrication and mechanical, optical and wettability characterization of the PDMS/beeswax composite. At this stage, experimental tensile test, hardness, wettability, spectrophotometric, dynamic mechanical analysis and thermogravimetric were carried out for the composite manufactured. The trials were handled by Origin software and Universal Ta instruments. The results were analyzed and discussed showing the improved properties and possible applications of this composite.

In the sixth chapter is described the general conclusion and relative to the stages of the work and described some proposals for future work.

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## Chapter 2

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# PDMS characterization and its applications: A review <sup>1</sup>

### Abstract

Polydimethylsiloxane (PDMS) is one of the most promising elastomers, as a material with good thermal stability, biocompatibility corrosion resistance, flexibility, low cost, ease of use, chemically inertia, hyperplastic characteristics, and gas permeability. Thus, it can be used in areas like microfluidic systems, medical devices, electronic components, membranes for filtering and pervaporation, sensors and coatings. Besides that, pure PDMS has low mechanical properties, as low modulus of elasticity and strength, which can be modified by mixing it with other polymers, adding particles or reinforcements. Fiber-reinforced PDMS proved to be a good alternative to the manufacturing of flexible displays, batteries, wearable devices, tactile sensors, and energy harvesting systems. PDMS and particulates are commonly used in the separation of liquids from wastewater due to porosity followed by hydrophobicity. Waxes as beeswax and paraffin proved to be materials capable of improving hydrophobic, corrosion resistance, thermal and optical properties of PDMS. Finally, blended with polymers such as poly (vinyl chloride-co-vinyl acetate), PDMS became a highly efficient alternative for separation membrane applications. However, there are few works dedicated to listing, review and compare information about different PDMS composites. So, this review article discusses this composite, their respective applications, and properties. The topics discuss the combination of elastomer with fibers, particles, waxes, polymers, and others; aiming to be a base text demonstrating the wide applications of this material and how tailored characteristics can be reached for custom applications.

**Keywords:** Polydimethylsiloxane (PDMS); Biocompatibility; mechanical and optical properties; Composite; Transparency.

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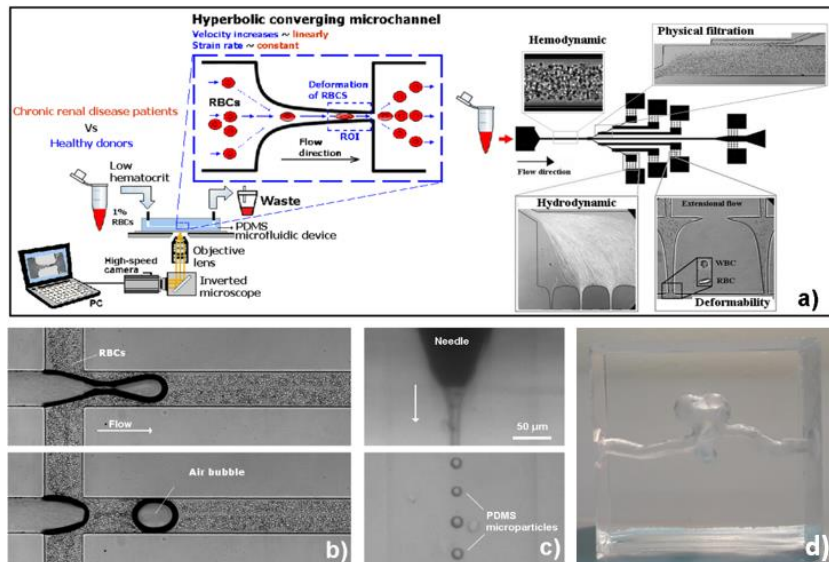
## 2.1 Introduction

Polymers are a large class of materials, widely used, and the basis of several industrial goods [1]. They are formed by the chemical linking of smaller molecules, the monomers, and, according to the number and characteristics of this bonded molecules, different properties can be achieved [2]. This way, they are used in diverse applications and can be found on daily use products like synthetic fibers, plastic bags, paints, lenses, glues and biomedical devices [3–8]. Also, the polymeric industry has grown quickly and nowadays is larger than aluminum, copper, and some other big material transformation industries [9].

Among this, an important subclass of materials is the elastomers. They are loosely cross-linked polymers, generally having low Young's modulus, supporting high yield strain and being able to restore the original shape when a stress, which caused the deformation, ceases (property known as elasticity). Examples include natural and synthetic rubbers, silicone elastomers, and other copolymers [10,11].

One of the most promising elastomers is the Polydimethylsiloxane (PDMS). It is a highly used silicon-based polymer, with good chemical and thermal stability [12–15], biocompatibility [16–18] corrosion resistance [19,20], flexibility [21–23], repeatability [24], low cost [25], ease of use, chemically inertia, hyperplastic characteristics and gas permeability [26–30]. Thus, it can be used in areas, such as: microfluidic systems [31–37], biomodels [38,39], organ-on-a-chip platforms [40], blood analogues [41–45], electronic components [46], membranes for filtering and pervaporation [47–49], sensors [50–52], optical and thermal devices [53–55], coatings [56–58], etc. Figure 2.1 shows few examples of PDMS applications in fields like microfluidic and biomedical.

However, pure PDMS applications can be limited due to its low mechanical properties, as low modulus of elasticity and strength. This can be solved by bulk modifications that create PDMS' composites with tailored properties, done by inserting free molecules, nano or microparticles, and by changing the pre-polymer composition [23]. Another works presented in literature also point out to tensile properties improvements when adding fibers [59].



**Figure 2.1- PDMS applications a) in microfluidic devices to assess of motions and deformations of red blood cells (RBCs) from healthy donors and pathological patients [32, 35]; b) in microchannel networks to investigate gas embolism [37]; c) with a flow focusing technique to generate micro-sized PDMS particles [43]; d) with PDMS biomodels to assess the blood flow behavior in aneurysms [38].**

Despite the extensive number of works that show applications and properties of pure PDMS, there are a few works dedicated to list, review and compare information about its composites. So, this review article discusses this material, their respective uses, and characteristics. Therefore, an overview of the vast literature on the subject was made, highlighting the works in which changes in the properties are reached by some other material addition. The topics discuss the combination of elastomer with fibers, particles, waxes, polymers, and others; aiming to be a base text to demonstrate the wide applications of this material and how customized characteristics can be reached for each of these applications.

## 2.2 PDMS

PDMS is a polymer classified as a silicon elastomer, which means it is constituted by a combination of inorganic chains with high surface energy, associated with silicates, and methyl groups, inorganics and with low surface energy. Inside PDMS' chemical structure, these methyl groups are prevalent to provide hydrophobic characteristics, with surface tension around 20.4 mN/m [60].

The chemical structure consists in a (Si-O) backbone and repetitive (Si(CH<sub>3</sub>)<sub>2</sub>O) units that can be expressed as CH<sub>3</sub>[Si(CH<sub>3</sub>)<sub>2</sub>O]<sub>n</sub>Si(CH<sub>3</sub>)<sub>3</sub>, whereas n represents the number of repetitive units. These repetitions define materials molecular weight, which consequently defines properties as viscoelasticity. Also, in these units, (CH<sub>3</sub>) represents the methyl group, and, the strength of siloxane (Si-O) bonds, makes this material chemical

and thermally stable. Crosslinking reaction, with groups as phenyl and vinyl can accomplish great properties changes, for different applications [23,61,62].

One of the main areas where this elastomer is used is the biomedical, thus the material presents high biocompatibility and biostability. These terms are related with materials that do not cause adverse effects in contact with biological tissues. Although the mechanism for biocompatibility still not totally clearly demonstrated, it is known that interactions with water in proteins are fundamental parameters and are related with physicochemical characteristics as surface free energy, stiffness, surface charge and wettability [63].

Another important aspect for biomaterials refers to structural biocompatibility. This is related with the mechanical interactions between an implanted device and the surrounding tissues. Mismatch of mechanical properties can cause inflammation or incorrect support of the efforts present [64].

Regarding mechanical properties, pure PDMS usually shows Elastic Modulus between 1.32 and 2.97 MPa and tensile strength from 3.51 to 5.13 MPa. These values variate according to the mixing ratio and curing temperature during the manufacturing process; being related that maximum the tensile values reached rise with the temperature rising until reach 125°C; above these value, higher temperatures reduce the tensile strength, but Young's modulus keep growing and presents a linear relation with the temperature. [65,66]. An increasing on the curing agent ratio can lead a reduction PDMS' flexibility and, consequently, reduces the Young's Modulus [67,68]. The hardness usually is proportional to the Young's modulus and presents values around 43 Shore A [69–71].

Besides being optical transparent, having low cost and high capability to replicate models or being used in rapid prototyping, pure PDMS has limitations. For instance, high hydrophobicity can be a problem when filling microchannels, being necessary to resort to temporary surface treatment procedures, as oxygen plasma. Other restraints occur due to the permeability of the material, which can interfere in cell cultures. Main disadvantages can be noticed as structural applications and may be extremely specific and reduced. Furthermore, the modification of characteristics, such as transparency, can be interesting for the use in sensors and some types of coatings [23,64,72].

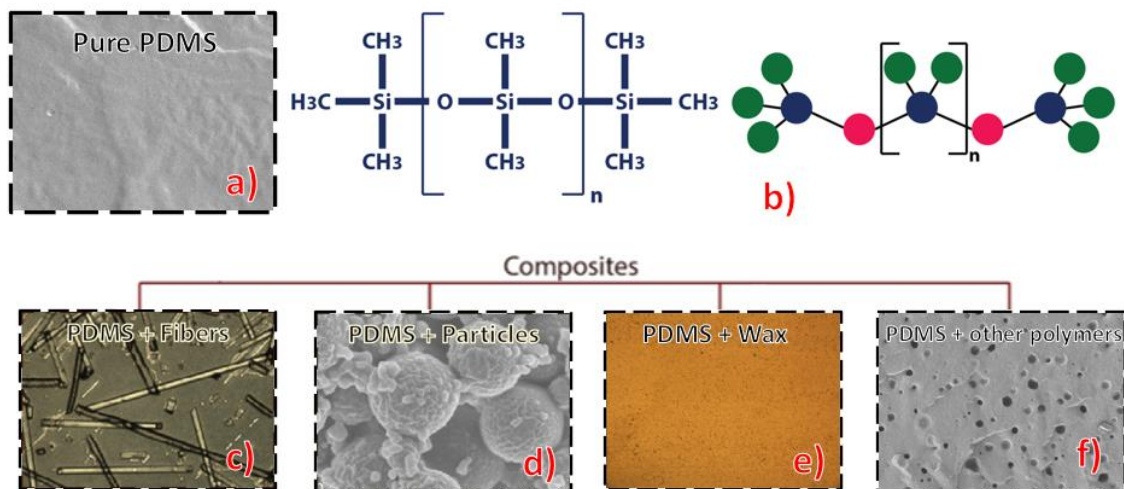
## 2.3 PDMS composites

Despite the large deformation capacity of the pure elastomers, fillers or reinforcements are often used to create composites that usually exhibit increased stiffness modulus, fracture toughness, fatigue resistance, tensile strength and abrasion resistance [73,74].

Thus, filler characteristics such volumetric fraction, shape, size and dispersion are extremely important [75].

Even more crucial for the final properties is the interaction between the elastomer and the filler, which increases the degree of crosslinking. This characteristic is optimized if there are reactive surface groups and the particles inserted are small, which is the reason why many of the reinforcements used in this type of composite are nano or microfibers and particles, see figure 2.2(c-d). Also, some agents can help the dispersion and coupling of the fillers on the composite. Usually, they are bifunctional molecules that provide bridges at the polymer/filler interface. Examples are silicone coupling agents, mercaptopropyltrimethoxysilane and (3-triethoxysilylpropyl) tetra sulfide [76,77].

Therefore, the combination of PDMS with other materials allows the optimization and expansion of its applications and has already been extensively explored. The next topics discuss some of the main composites with PDMS matrix, dividing them into materials filled with fibers or particles, and materials in which there is a combination with waxes (figure 2.2e), polymers (figure 2.2f) or another additive.



**Figure 2.2-** Pure PDMS and its composites discussed in the present work: (a) Pure PDMS, (b) Chemical composition; Composites: (c) with Glass Fiber Reinforcement, image adapted from [59]; (d) with tantalum ethoxide – nanoparticles, image adapted from [78]; (e) with beeswax; (f) with other polymer combination (tetraethoxysilane (TEOS)), image adapted from [79].

### 2.3.1 Fiber and nanofiber reinforced PDMS

Fiber reinforced composites are one of the most successful materials in engineering applications nowadays. Besides the matrix usually being polymers as epoxy or polyurethane, the use of elastomer matrices can be promising for applications as “muscle” actuator, flexible surfaces for aircraft aerodynamic components and safer flywheels [80]. Also, for biomedical applications, carbon fillers inside a polymer can become an alternative to metallic devices as they present advantages as being radiolucent and have high mechanical properties. However, after cyclical sterilization, the interaction between phases can be compromised [81].

Likewise, fibrous fillers for PDMS are not widely studied in the literature. The distribution of reinforcements can be difficult, especially when using nanofibers, generating fillers' agglomeration and voids, which impairs the properties and complicate the mechanical. However, studies in which electrospun polyacrylonitrile-graft-polydimethylsiloxane fibers were introduced as a graft copolymer on a PDMS matrix in non-woven and aligned portions, reached satisfactory results, increasing the tensile strength from 0.3MPa up to 2.3MPa. Also, the Young Modulus that was 47MPa for the pure silicone sample, became 119MPa, when using non-woven fillers, and 674MPa, for aligned reinforcements [82]. Another work reached an increase in mechanical and thermal properties when using a PDMS and graphene foam matrix reinforced with carbon fiber. This increasing, in the tensile strength, Young's modulus and thermal conductivity was, respectively, 52%, 71%, and 41%, when comparing with the pure matrix [83].

A combination of PDMS with carbon fillers has already been proposed as an alternative for the fabrication of flexible displays, batteries, wearable devices and tactile sensors, used for robotics [49,50]. By combining short carbon fiber with elastomer in a method known as Spatial Confining Forced Network Assembly, it was obtained electrical conductivity around  $1.67 \cdot 10^2$  S/m, superior to that presented by pure PDMS, an insulator with conductivity in the order of  $10^{-12}$  S/m. Also, when pre-compressed, this composite can reach even higher conductivity, around  $3.2 \cdot 10^2$  S/m [84].

When using dual-scale carbon fillers, a combination of carbon nanofibers with short carbon fibers, the disparity between the elastic modulus of the elastomeric matrix and the conductive graft, used as a sensor, was reduced. This leads to increased sensitivity, stretchability and repeatability of high-strain sensors, reaching small drifts in resistance even after 300 loading cycles [85]. A related application is in harvesting energy

devices manufacturing. These normally are constituted by a piezoelectric ceramic and present problems like the possibility of failures when submitted to cyclical efforts, due to the fragility of the ceramic, and environmental impact as they usually contain lead, being classified as Substance of Very High Concern.  $K_{0.485}Na_{0.485}Li_{0.03}NbO_3$  (KNLN) fibers in PDMS have achieved piezoelectric charge and voltage comparable to that of ceramic PZTs, in a mechanically compliant and environmentally friendly material [86]. Satisfactory results for energy harvesting and piezoelectric uses were also assessed using the elastomeric matrix and  $BaTiO_3$  nanofibers, another lead-free alternative that can be aligned in the matrix and reached output voltage values between 0.56 and 2.67V by applying a periodic mechanical compression with a pressure of 2Pa [87]. Also, using silver micro fiber in a soft lithography process, the conductivity of the PDMS increased, changing the resistance from 0.12  $\Omega m$  (for 10% wt) to 0.000001  $\Omega m$ ; using 8% wt of short carbon fiber fillers, the resistance of a 0.1 mm thick sample was 0.0026 and could be reach to values 3 times lower with the insertion 1% wt of a second conductive nanofiller [88,89].

Reinforcements can also allow the use of PDMS in high-performance composites, maintaining good transparency. Studies with just 1% wt of three-dimensional silica continuous fiber reinforcements achieved, in comparison with the pure matrix, increases of 140%, 94%, 18% and 95% in tensile modulus, strength, maximum strain and tear strength, respectively [59]. The optical properties of the elastomer can also be explored to analyze fiber alignment in processes such as the injection molding, where a study was able to visualize the flow during the injection and notice how carbon fiber inserts positioning could be improved [90]. Furthermore, the use of PDMS as NCF fiber optic interferometer coating showed excellent thermal performance, resulting in a significant improvement in temperature sensitivity when compared to pure silica fiber interferometer. PDMS was used as fiber coating improving the fragility of the fiber structure, thus increasing its reliability for practical applications [52]. Likewise, tailored properties can be achieved to match living tissue requirements, as bone elasticity modulus. This way, the combination 55 vol.% of aramid balanced fabric reinforcement, PDMS and 0-25% vol.% nano/micro hydroxyapatite and tricalcium phosphate reached modulus of elasticity, in bending, similar to that presented by human cortical bones (14-20GPa) being suitable in bone surgeries [91]. For stretchable electronics, a PDMS/plain weft-knitted nylon fabric showed a huge increase in fracture toughness, about 700%,

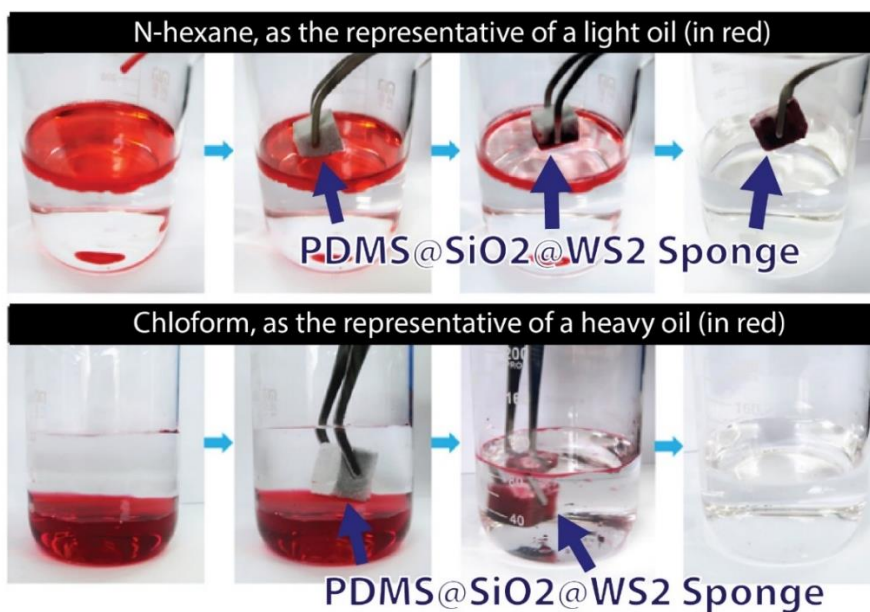


much higher than of pure PDMS and keeping high elasticity (Young's modulus around 2MPa). Also, during cyclic loading, for small stretches, the behavior was linear elastic and, for a relatively large stretch, it has significant hysteresis [92].

### **2.3.2 Addition of particles to PDMS**

Particles are one of the most common additions in PDMS. When one of the dimensions of the inserted fillers is smaller than 100nm, the formation of nanocomposites occurs. Another common occurrence is micro composites, with particles on the micrometric scale. Examples of particles are carbon nanotubes, some silicates and graphene [23].

Porous compounds using PDMS and particulates are commonly used to improve absorption efficiency in the separation of liquids from wastewater due to their porosity and super hydrophobicity. PDMS shows benefits for the reusable compound, as can be pressed repeatedly without structure loss. Also, as a low-cost material, it can reduce the cost of effluents treatment as these processes generally requires a large number of absorbent materials. Nanoparticles are used to improve surface roughness, which leads to an increase in super hydrophobicity, reaching a contact angle with water greater than 150° and making the droplets slide under the surface. PDMS acts as a strong binding and immunization adhesive for nanoparticles and one of the main advantages of this application is its non-toxicity. Some works manufactured porous compounds modified with SiO<sub>2</sub> nanoparticles and others with TiO<sub>2</sub> nanoparticles, coated with PDMS and manufactured by a simple immersion technique. Melanin sponges have high efficiency removing oil from water through micro and nanoparticles of tungsten disulfide (WS<sub>2</sub>) and SiO<sub>2</sub>, respectively. The particles are immobilized on the sponge surface by a layer of polydimethylsiloxane adhesive that features an extremely water-repellent structure by a simple one-step immersion process. The composite sponge has high oil absorption with 21e112 times its own weight and a selectivity efficiency above 99.8%, as shown figure 2.3 [93]. Likewise, using silicon nanoparticles as a coating substrate was produced by simple immersion followed by magnetic stirring in order to reduce the surface drag force. The drag force reduction rate decreased up to 24% and the coating showed greater durability in acidic and alkaline solutions [94]. TiO<sub>2</sub> additives are also applied to create electroactive material based on PDMS, this shows reduced drive voltage and response speed in comparison with a traditional Silicone-Poly(hexylthiophene) electroactive polymer[95].



**Figure 2.3- PDMS/SiO<sub>2</sub>/WS<sub>2</sub> sponge for application in oil separation, adapted from [93].**

The metallic meshes with different pore sizes were coated with nanoparticles and nanocomposites by an immersion process. Hydrophobic titanium diode was used as a nanoparticle and polydimethylsiloxane as a binding resin. The presence of PDMS resulted in an improvement in the mechanical durability of wire mesh. Besides that, it was found that meshes with smaller pores are more efficient in separation, however the process takes longer [96]. Likewise, porous sponge with photocatalytic properties, manufactured with PDMS and TiO<sub>2</sub>, was used for the degradation and demolition of organic pollutants in textile wastewater. These nanoparticles are injected into the polydimethylsiloxane sponge that becomes able to demote up to 50% of the pollutants without the presence of light and 80% with light, this is due to degradation to the photocatalytic action of TiO<sub>2</sub> allowing greater absorption of the dye from the solution [97]. As well, TiO<sub>2</sub> nanoparticles were added to the PDMS matrix by a spin coating method and used as coatings on metals to improve the corrosive capacity of the material. Corrosion resistance improved with the 8% by weight proportion of TiO<sub>2</sub> that achieved the best performance [98].

In addition, metallic particles in PDMS films or substrates have been studied for various applications. For example, micro-pumps can be created, taking advantage of the elasticity and mobility of PDMS membranes, and using magnetic actuators to generate deflections by attracting iron particles [99]. The same composite with magnetic properties could find application in the control of droplets motion, a useful resource in the manipulation of sample drops in chemical and biological tests and studies [100]. When comparing different magnetic fillers, pristine carbonyl iron microparticles (CI) reached a

maximum deflection of 762  $\mu\text{m}$  at 0.27 mT for a membrane diameter of 6.2 mm, which was the highest result in a study that also evaluated lauric acid-coated superparamagnetic iron-oxide nanoparticles (SPION-LA) and lauric acid-coated carbonyl iron microparticles (CI-LA) [101].

The porosity and water absorption of the PDMS cell scaffolds are inversely proportional to particle size. However, the interconnectivity of PDMS cell scaffolds increases with increasing in the particles dimension. Also, mechanical properties such as compressive modulus and compressive strength obtained higher values for intermediate pore sizes, between 300–450  $\mu\text{m}$ , in a sample universe with pores of 150–300, 300–450, and 450–600  $\mu\text{m}$  [102].

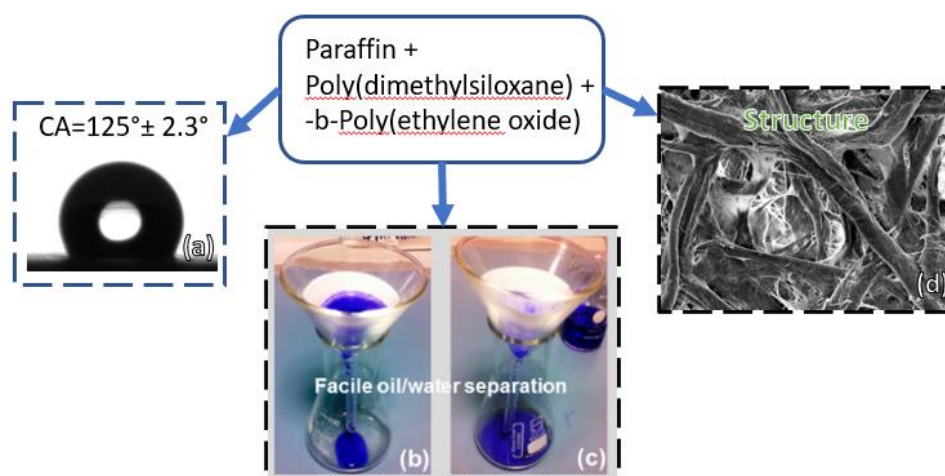
### **2.3.3 Wax Addition**

Waxes as beeswax and paraffin proved to be materials capable of improving hydrophobicity corrosion resistance, thermal and optical properties of PDMS, leading to applications as wearable devices, sensors and superhydrophobic coating [72].

A phase change functional compound consisting of PDMS as matrix and paraffin was prepared by the molten mixture method and has excellent performance in thermal and mechanical properties. The compound has excellent flexibility and heat absorption capacity. As the paraffin content increases, the temperature sensitivity of the mechanical property also increases and the composite's storage modulus decreases with increasing temperature. Consequently, substrate thermal management flexibility will be enhanced with increasing temperature, thus applying to a flexible substrate. The change in the transmittance of composites due to the phase change of the paraffin makes it applicable as a visual warning of temperature increase [103]. Also with the addition of paraffin spheres in PDMS matrix, the composite has an increase of more than an order of magnitude in stiffness in a compression test [104]. Another work was studied to improve the transparency of the compound with a percentage of up to 10% wt of paraffin without changing the transparency of the film. The compound has demonstrated after optical testing that the material is not ideal for use as a transparent reflective key, but there have been considerable additional improvements [105].

Another interesting coating using PDMS combined with paraffin wax is in textile engineering manufactured by a simple an inlaying process. The prepared textiles exhibited stability after mechanical abrasion and chemical corrosion. Furthermore, the coated textiles have excellent self-cleaning ability, water repellence ability and can be

used to separate various types of liquid mixtures such as oil-water, diesel oil and crude oil. The separation efficiency can reach up to 95% in separation of the diesel oil/water mixture specifically [106]. A new method of modifying the surface properties of filter papers making them super hydrophilic and super oleophobic underwater in a simple and economical way is presented. Both super hydrophilic and super oleophobic paper have excellent efficiency for gravity oil-water separation, being up to 99% [107]. Figure 2.4 shows the experimental of oil/water separation by filter paper, evaluating the wettability and showing the structure of filter paper.



**Figure 2.4- Oil/water separation by filter paper, oil remains on the filter paper while water with blue dye passes through the filter paper, adapted from [107]: (a) Contact angle, (b, c) oil/water separation and (d) structure of filter paper.**

Other work of transparent and flexible composite films with selectable mist by a lamination process were manufactured. The film is composed of polyethylene terephthalate, graphene, paraffin organogel-PDMS substrates. When graphene is heated, a transformation occurs in the paraffin impregnated in the PDMS, leading to an improvement in light scattering. Transmittance is maintained above 90% in the visible range, while fog can be controlled in the range of 0.5 to 85% by applying a voltage of 18V with a consumption of 0.33W/cm<sup>2</sup>. Optical film that enables transmittance control can be used to enhance the light capturing properties for photovoltaic panels and windows, allowing privacy [108].

Another way is to apply a voltage of 10V on the composite film showing rapid change between opaque and transparent in less than 8s followed by transmittance which changes from 2% to 75% [109].

However, it is also possible to have adhesive films with the same function of switching between opaque and transparent via a thermal trigger. The appearance of the films alternates between opaque at room temperature and clear at temperatures above 53°C. The change in optical properties is almost instantaneous and the application is best suited for use as a low-cost, smart window coating [110].

In order to meet the needs of protection against oxidative substances, corrosive liquids and ultraviolet light that limit the practical application of solar vapor generation. Photothermal conversion coatings were manufactured with stable chemical and mechanical properties by a spray process of a mixture of beeswax, multiwalled carbon nanotubes and polydimethylsiloxane. The coatings exhibited good broadband light absorption capacity efficiently under sunlight irradiation. The superhydrophobicity caused by beeswax and PDMS provides self-cleaning that can prevent the reduction of steam generation efficiency induced by microorganisms and mud in the water and are able to heal damage to superhydrophobicity through the migration of beeswax, providing lasting protection. Due to their low maintenance requirements, simple preparation process and high cost-effectiveness, photothermal conversion coatings can be suitable for supplying fresh water to remote/disastrous areas [111].

Another work used natural carnauba wax and PDMS to fabricate superhydrophobic surfaces by model transfer and colloidal deposition method, which consists of depositing carnauba wax on the surface and the secret to the superhydrophobic surface was rubbing to remove the wax particles weakly connected. After removing the loosely bound particles, the surface becomes super-hydrophobic with a contact angle greater than 150° and a slip angle less than 10°. The surface has good mechanical durability against abrasion and water impact. As they are biocompatible materials and with these characteristics, they have become widely used in biomedical applications with repellence to blood and its components, also presenting reduced drag to the blood and coagulation time of at least one hour [112]. This makes it ideal for manufacturing blood-compatible materials, microfluid devices used in blood separation and typing, and application to self-cleaning surgical garments. The super hydrophobic surface containing carnauba wax and PDMS has good mechanical durability against abrasion and water impacts, therefore suitable for outdoor environments [113].

Another application of PDMS combined with paraffin wax is in a new hermetic encapsulation method for microfluidic devices actuated by negative pressure. The airtight

materials used are non-active, non-hazardous, and commonly used as sealing materials for plastic medicine and food packaging. The advantages of the new method are providing the ability to steer for more than 3 weeks without any vacuum equipment and that the air intake is only when the device is encapsulated [114].

#### **2.3.4 Blends with other polymers**

The search for ecological materials for separation membranes is becoming highly attractive and increasingly competitive, always focusing on low cost with high separation efficiency that is present in the main industrial processes. New gas separation membranes using a copolymer of poly(vinyl chloride-co-vinylacetate)(PVCA) and polydimethylsiloxane(PDMS) were manufactured by a simple method of mixing followed by constant agitation. The separation performance was done by permeation studies of pure phases CO<sub>2</sub>, N<sub>2</sub> and CH<sub>4</sub> at different temperatures. After characterization tests, the membranes showed better selectivity and high flux at 25°C and as the temperature increases up to 75°C, the selectivity and flux decreases. It was found that with the addition of PVCA to PDMS, the mechanical and thermal stability improved by more than 25% and 6%, respectively. The results were positive from the combination of PVCA and PDMS consisting in the development of a homogeneous dense film structure [115]. Likewise, PDMS-graft copolyimides were synthesized by polycondensation followed by chemical imidization to investigate the effects of the PDMS segment, and then the copolyimide membranes were prepared by the solvent casting method. The gas permeability coefficients of the copolymer membranes increase with the increase in the length of the PDMS segment, but they decreased after heat treatment at 200°C. In addition, high gas permeability caused by the continuous phase of the PDMS flexible domain along with improved pervaporation when it requires efficient removal of VOCs from aqueous mixtures was verified. PDMS grafted copolymer membranes show efficiency in removing toxic organic components from wastewater due to improved pervaporation technique [116]. Also, another pervaporation membrane composed of SiO<sub>2</sub>/PDMS/PVDF was manufactured by the dynamic negative pressure method and showed significant improvement in the contact angle with the surface by more than 40% compared to the PVDF membrane alone. Permeation of phenol over water increases. The SiO<sub>2</sub> dispersed in the PDMS solution improves mechanical strength and phenol recovery which strongly suggests the removal of phenols in wastewater from coal gasification [117].

With the aim of making the pervaporation process more efficient and on a large scale of production, a non-porous PDMS composite membrane with surface standardization in two stages was manufactured. First, patterning the PVDF substrate in two layers using phase separation micro-molding followed by modified dipping precipitation, and then, the PDMS solution was prepared with different types of crosslinking agents were cast onto the patterned substrate prepared as selective layer. Permeation of the standardized membrane with the crosslinking agent TEOS had a larger pattern size, generally more than 2 times higher than the non-standardized one, while with VTES and p-TTES they improved the polymer stiffness thus it is an effective way to improve the pervaporation flow. Micropatterned PDMS composite membranes will show great potential in large-scale industrial application for bioethanol recovery [58].

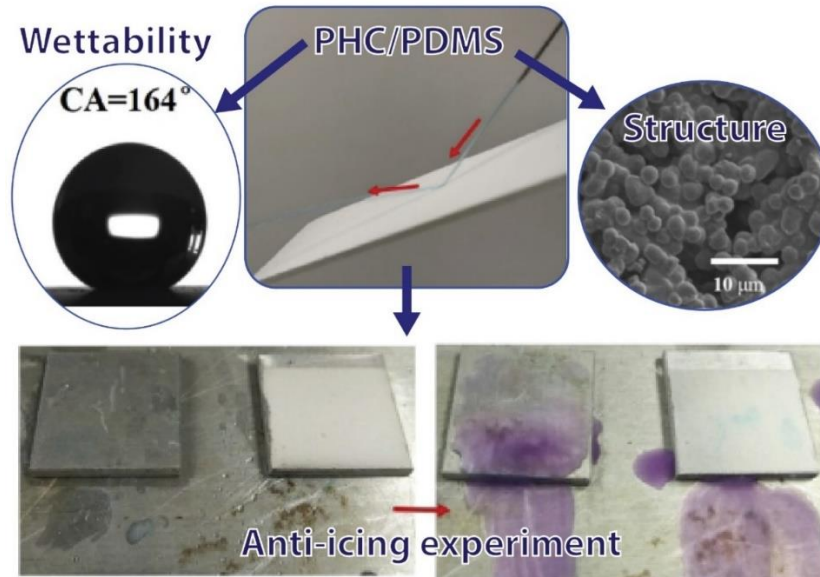
In the investigation of solutions to ice accumulation on surfaces, superhydrophobic coatings using crosslinked PHC microspheres that have been combined with adhesive PDMS by a single-step precipitation polymerization process are presented. The coated surface showed high contact angle, good mechanical durability due to micro/nanoscale structure, self-cleaning, and excellent water, and ice repellent properties at low temperatures. Its applications are promising and efficient for anti-freeze coatings on external structures, as shown figure 2.5 [118].

In several fields of application in biomedicine, PDMS stands out for being a biocompatible, non-toxic, transparent material with good thermal and mechanical properties, considering the low cost of manufacturing and raw material. Elastomer PSUs were prepared from amino propyl terminated PDMS, H12MDI and APTMDS by a two-step polyaddition route. The PSU films showed high transparency above 90% in the visible region, low elastic modulus, the hysteresis values decreased from 32 to 2% in the tenth cycle and the soft segment refractive index was increased through the incorporation of 14 mol% of methyl-phenyl-siloxane. These results reveal that it is possible to use PSU films to replace the natural human lens after cataract surgery [119].

### **2.3.5 Other additions**

Another option is emerging in transforming surfaces of different types of substrates into super hydrophobic surfaces using the phase separation method. PDMS is used as a binder, tetrahydrofuran (THF) as a solvent and water as a non-solvent. The modified fabric showed good self-cleaning ability, antifouling and excellent superhydrophobic property with a contact angle above  $150^\circ$  and a slip angle below  $10^\circ$  which

are ideal conditions for oil-water separation devices. The advantages of this method are that there is no addition of nanoparticles, thus leaving no marks and no change in tissue color. The easy synthesis method has a wide application potential for fabrication of super hydrophobic surfaces [120].



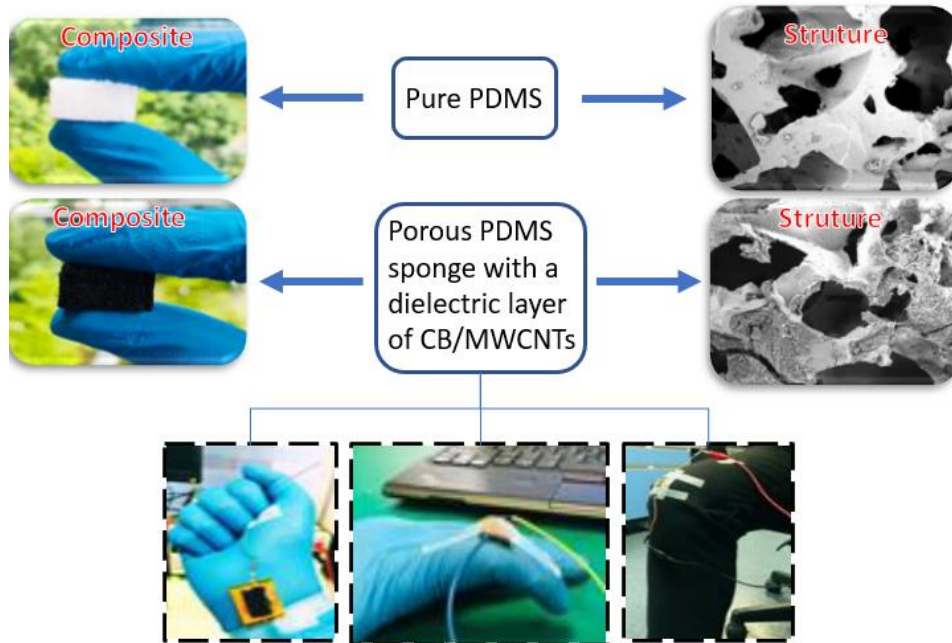
**Figure 2.5- Using PHC/PDMS blends for anti-ice application, adapted from [118].**

Polyethylene glycol blended polydimethylsiloxane elastomeric films were manufactured by a simple mixing process followed by mechanical agitation. The elastomeric films showed an increase in the degree of swelling as the amount of PEG increases, and the young modulus decreases with an increase in the amount of PEG. Although the effect of PEG on PDMS crosslinking deteriorates the mechanical properties of the material, it can be considered positive in terms of increasing hydrophilicity [79].

Flexible tactile sensors based on three-dimensional (3D) porous conductive composites were designed with a homogeneous synergistic conductive network of carbon black (CB) and carbon nanotube (MWCNTs) single dip coating on a polydimethylsiloxane (PDMS) sponge skeleton, as shown figure 2.6. The 3D porous structure with hybrid conductive networks of CB/MWCNTs exhibited superior elasticity and excellent electrical characterization under external compression. This piezoresistive tactile sensor exhibited high sensitivity ( $15 \text{ kPa}^{-1}$ ), fast response time (100 ms), ability to detect small and large compressive deformations as well as mechanical deformability and stability over 1000 cycles. The piezoresistive sensor has been used successfully in monitoring human physiological signals including finger heart rate, pulses, knee flexion,



breathing, and finger gripping movements. The highly sensitive piezo-resistive sensor indicates great potential for applications in robotic-assisted surgery system, human-machine interface and low-cost wearable health electronics [121].



**Figure 2.6- 3D porous sponge made of PDMS, carbon black and carbon nanotube for application in monitoring physiological signals, breathing, gripping movements and heart rate, adapted from [121]**

Membranes composed by polydimethylsiloxane combined with other additives have been studied in the most diverse everyday applications. The PDMS-D2HPA compound forms a gel layer on the outer surface of PVDF ultrafiltration hollow fibers by a gellification technique in application to a new gel extraction membrane (EGM). Optimal extraction efficiency and EGM stability were achieved. The composite membrane showed improvement in long-term operational stability than EGM flux attenuation by only 34% in 120h, whereas the conventional SLM was 100% in 45h. The advantages were evident, showing greater flow and operational stability of the EGM over the conventional SLM process [122]. However, unlike the electrospun PVDF layer in PDMS-coated membranes, the electrospun PDMS/PMMA or PDMS/PMMA/TPU membranes can be applied directly in the separation of wastewater from saline phenol with better mass transfer coefficient [12].

Another work related to the selection of liquids consists of a porous hydrophobic sponge made of polydimethylsiloxane incorporating a small amount of graphene on the PDMS sponge skeleton surface by a process of mold transfer - surface incorporation. The

graphene embedded PDMS sponge showed improved elasticity and durability, followed by less time needed to absorb the same amount of oil. However, robust and elastic mechanical sponges can be produced for direct application in oil/water separation in an underwater environment with less absorption time [123].

With the strong demand for superhydrophobic and self-cleaning surfaces, coatings containing PDMS and APTES were manufactured by a simple immersion process followed by room temperature curing. The coatings exhibited high contact angle, high transparency in the visible region, followed by excellent self-cleaning properties indoors and outdoors. In addition, the hybrid coating exhibited excellent antifogging behavior after prolonged exposure to mist and also presented good drop impact durability when applied outdoors [124]. In order to improve surfaces for superhydrophobic and self-cleaning properties, the PDMS-modified PU/Al coating with a smooth surface was prepared using PDMS-modified PU and Al powder flake as resin matrix and functional pigment. However, when the PDMS-modified PU/Al coating was modified by nano-SiO<sub>2</sub> it presented a distinct micro-nano mastoid structure that was formed on the surface allowing to reach a contact angle above 150° and a slip angle of less than 10°, thus, obtaining excellent self-cleaning performance and super-hydrophobicity [125].

Crosslinked membranes were fabricated by introducing LCs into a PDMS matrix by the preferred crosslinking method. The crosslinked membranes produced showed increased mechanical properties compared to pure PDMS membranes. PDMS/LC membranes also showed better membrane formation capacity, lower hemolysis rate, lower platelet adhesion and more favorable anticoagulant properties. Furthermore, the mechanical properties and blood compatibility of the membrane may be increased due to the introduction of cholesteric liquid crystals [17].

Surfaces with charge density, roughness and morphology have a strong influence on the interaction of biomaterials and cells. A hybrid coating with hierarchical surface structures consisting of PDMS and tantalum oxide were fabricated by a simple one-pot sol-gel-based method. The hybrid coatings showed structures with a combination of micron/submicron and nanoscale characteristics. Structures can be tailored from porous multilayer to single layer films, depending on the concentration of tantalum oxide. The coatings showed good fibroblast adhesion and cell proliferation as a demonstration of their ability to modulate cell functions. This study has a potential application in the biomedical field to modulate cellular responses and improve implant performance [78].

Another field of application for PDMS is in a multifunctional dressing material, introducing a series of reduced graphene oxide (rGO) sheet contents into the PDMS matrix. The porous membrane was fabricated using the solvent evaporation induced phase separation technique. The high porosity of the rGO-PDMS membrane on the lower surface is beneficial for cell adhesion and proliferation, and the small pores on the upper surface can prevent excessive water loss in wounds. The addition of rGO blades to the PDMS has improved mechanical strength and can be applied to dressings under high tension. The rGO-PDMS composite membrane showed an increase of 35.33% in mechanical strength and 34.38% in elastic modulus. The membranes also showed inhibition of bacterial growth and remarkably accelerated wound healing through increased re-epithelialization and formation of granulation tissue. Therefore, a promising material that can be considered a multifunctional dressing [7].

## 2.4 Results Summary

Table 2.1 describes the main applications of PDMS mixed with other different types of materials, allowing the analysis and comparison of some properties directly and indirectly. However, there is a summary of what was written in the body of the article with some mechanical, optical and wettability properties.

**Table 2.1- Summary of PDMS properties combined with other materials.**

Type	Description	Reinforcement	Property	Value/change	References
Additive	Membrane	PDMS-PEG1	Young's modulus	Decreased 22.0%	[79]
			TS	Decreased 6.0%	
Additive	Sponge	Graphene/PDMS	WCA	128.9 ±2.3°	[123]
Additive	Membrane	PDMS-D2EHPA	WCA	102.0±2.0°	[122]
Additive	Sponge	THF as the solvent	WCA	155.0±0.6°	[120]
Additive	Membrane	PDMS/PMMA	TS at break	1.7MPa	[12]
			Elongation at break	60.0%	
Additive	Membrane	rGO-PDMS	TS	Increased 35.33%	[7]
			Young's Modulus	Increased 34.38%	
Additive	Membrane	PDMS/LC	Strain at break	Increased 7.0%	[17]
			Stress at break	Increased 78.0%	
			Elastic modulus	4.7MPa	
Additive	Coating	PDMS/APTES	WCA	103.9°	[124]
			Transparency	91.2%	
Blend	Membrane	PDMS/PVCA	UTS	133.7MPa	[115]
			Elastic modulus	2400MPa	

Blend	Membrane	PIS6	WCA	99.2°	[116]
			Young's Modulus	400MPa	
			TS	20MPa	
			Elongation at break	100%	
Blend	Characterization	PSU-3T	Young's Modulus	5.5MPa	[126]
			UTS	6.0MPa	
			Elongation at break	880%	
Nanocomposite	Coating	PDMS/TEOS	WCA	130.0°	[127]
			WVP	$7.9 \times 10^{-8} \frac{\text{g}}{\text{msPa}}$	
Nanocomposite	Films	PDMS-clay	Elastic Modulus	1.5MPa	[128]
Nanoparticle	Coating	Tantalum oxide /PDMS	WCA	110.0°	[78]
Nanoparticle	Coating	PDMS/PU-Al and SiO2	WCA	151.5°	[125]
			SA	9.0°	
Nanoparticle	Coating	PDMS/ Spray-coated CNP	WCA	167.0°	[129]
Nanoparticle	Sponge	PDMS/SiO2/WS2	WCA	158.8±1.4°	[93]
			Separation Efficiency	99.8%	
Nanoparticle	Coating	SiO2/ PDMS and Beeswax	WCA	154.6°	[94]
			SA	5.0°	
Nanoparticle	Coating	PDMS/TiO2	WCA	102.0°	[98]
			Properties	Improved the anticorrosion	
Nanoparticle	Mechanical Properties	MQ resin in silica sol and V-PDMS	Young's modulus	0.2MPa	[130]
			TS	1.9MPa	
Nanoparticle	Coating	PDMS/TiO2	WCA	158.0°	[96]
			SA	5.0°	
			Separation Efficiency	Oil/water 99.5%	
			Properties	Improved Abrasion Resistant	
Particle	Coating	PDMS/PHC	WCA	164.0°	[118]
			SA	3.7°	
			Properties	Improved Mechanical Durability	

Particle	Membrane	PDMS/SiO <sub>2</sub> /PVD	WCA	131.8°	[117]
		F	Elongation at break	158.0%	
Particles	Membrane	PDMS-silicate-1	WCA	135.2°	[49]
Wax	Coating	PDMS-MCNTs-	WCA	158.3°	[111]
		Beeswax	SA	1.4°	
Wax	Coating	Carnauba	WCA	169.0°	[112]
		wax/PDMS-paper	SA	3.0°	
Wax	Multifunctional	PDMS/Paraffin	Transparency	80%	[104]
	Material				
Wax	Multifunctional	PDMS/Paraffin	Transparency	85.5%	[105]
	Material				
Wax	Multifunctional	P-PDMS	Transparency	~94.0%	[108]
	Material				
Wax	Multifunctional	PDMS/Paraffin	Transparency	85.0%	[110]
	Material				
Wax	Coating	Carnauba	WCA	162.0°	[113]
		wax/PDMS	SA	10.0°	
Wax	Coating	PDMS/Paraffin	WCA	156.7°	[106]
			Separation	Diesel oil/water	
			Efficiency	95%	
Wax	Mechanical	PDMS/Paraffin	TS	2MPa	[103]
	Properties		Transparency	~99%	
Wax	Mechanical	PDMS/Beeswax	WCA	129.3°	[72]
	Properties		TS	1.1MPa	
			Hardness	28 [Shore A]	
			Transparency	71%	
Wax	Mechanical	PDMS/Paraffin	WCA	141.9°	[72]
	Properties		TS	2.6MPa	
			Hardness	33.2[Shore A]	
			Transparency	72%	
Fiber	Mechanical	Poliacrilonitrila-	Young's modulus	Increased 56%	[82]
	Properties	graft-PDMS	Tensile strength	Increased 60%	
				non-woven	
Fiber	Mechanical	graphene	Young's modulus	Increased 71%	[83]
	Properties	foam/PDMS	Tensile strength	Increased 52%	
Fiber	Mechanical	Silica continuous	Maximum strain	Increased 94%	[59]
	Properties	/PDMS	Tensile Strength	Increased 140%	

According to the results presented in the table, PDMS composites modify the mechanical, thermal and surface wettability properties of PDMS. The additive-type studies by Zieh Pan et al.[123], Xiashi Ren et al. [122] and Gao Shouwei et al. [120] show

that addition composites helped to increase the surface hydrophobicity of PDMS sponges and membranes, thus improving the effectiveness for filtering oil in water and extracting heavy ions. Other work A. Syafiq et al. also modified the surface of PDMS to increase hydrophobicity and fabricate a self-cleaning glass substrate [124]. The works by Wei Qian et al. [7], Huaxin Rao et al. [17] and Long-Fei Ren et al. [12], sought to improve and improve the mechanical properties of PDMS by adding materials such as reduced graphene oxide (rGO), liquid crystal (LC) and PMMA sheets, for different applications such as biomedical in skin dressing and in direct application in separation of waste water. The additive-type PDMS composites presented, seek not only to improve mechanical properties and increase surface hydrophobicity, but also seek to reduce costs, proposing efficient and simple operation and manufacturing methods.

Some other types of materials reported are polymeric nanocomposites. Amir M. Nazari et al. [128] with the objective of manufacturing thin films to be applied in microfluidic devices, reinforced PDMS with nanoclay platelets and, by increasing the nanoclay content, it improved the elasticity of PDMS, but decreased the shear strength. Yanbing Luo et al. [127], used tetraethoxysilane (TEOS) nanocomposites to provide hydrophobic properties to sandstone and thus preserve it. In general, nanocomposites have physical properties superior to conventional composites, in terms of strength, rigidity, thermal and oxidative stability [128].

The vast majority of studies with nanoparticles were carried out to increase the hydrophobicity of PDMS [93,95,97,125,129], transforming the surfaces of coatings and sponges into super-hydrophobic, ie, the angle of contacts with water is greater than 150°. These are used for different applications such as self-cleaning surfaces, low infrared emissivity, adsorption (separation) of oil in water. However, in some studies [78,98] the hydrophobicity of PDMS was maintained, changing only the roughness[78] and its applications in anti-corrosive coatings [98] and in implants [78]. In a general context, for some applications the disadvantage of composites with nanoparticles is the loss of transparency. Similarly, studies carried out with particles changed the hydrophobicity of PDMS, increasing its value, the study by shiquan hang [118], which used poly[hexafluorobisphenol A-co-cyclotriphosphazene] (PHC) to create superhydrophobic coatings with angle of contact with 164° water, in order to have a self-cleaning and anti-freezing surface with mechanical properties and durability in aggressive

environments. However, this type of composite is not suitable for applications where the transparency of PDMS is required.

PDMS composites with different types of waxes mostly alter hydrophobic, transparency and mechanical properties. One of the characteristics so far not noticed in previous studies and observed in wax composites with PDMS, is the possibility to control the transparency with increasing temperature. In works that use paraffin, the composite films at room temperature remain opaque, however, as the temperature increases, the composite becomes transparent, reaching 99% transparency [43], in addition to the use of paraffin alters the hydrophobicity of PDMS making it superhydrophobic [106], another concern demonstrated with this type of composite is the use of biocompatible materials such as carnauba [112], so that the biocompatibility characteristics of PDMS are not changed, which enables the use of the composite in applications biomedical.

Finally, fiber composites with PDMS matrix reinforce the mechanical properties of PDMS, the research Hao-Yang Mi [59] uses silica fibers that increase the tensile modulus and strength by 140% and 94% respectively, with a loss of 20% of transparency. We can also note that this type of composite can improve thermal properties, as in the composite with graphene foam, short carbon fibers and PDMS, this composite improved thermal conductivity by 41%, tensile strength 52% and Young's modulus in 71% [83] compared to pure PDMS.

## **2.5 Conclusions**

PDMS has become a widely used material in many research fields, and its range of applications is increasing every year. This increase is reflected in the large number of works that have been studying and modifying this material, resulting in tailored properties for extremely specific purposes. In this review, it was provided an outlook how PDMS composites could have their main properties improved, such as mechanical, electrical, and optical features, opening new avenues and applications at various fields of engineering. The next generation of PDMS composite materials, should focus in improving and standardizing the manufacturing of PDMS mixed with other materials, such as waxes, and look for efficient processes that are easier to manufacture, with low cost, which allow the replacement of conventional processes and have large-scale engineering application. Additionally, PDMS can be widely explored in new emergent

technologies, as in the field of transparent films for photovoltaic panels, which continues to be a future trend regarding sustainable and renewable energy.

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## Chapter 3

# Multifunctional low-cost vacuum chamber to manufacturing PDMS based composites <sup>1</sup>

### Abstract

Polydimethylsiloxane (PDMS) is one of the most well-known elastomers and has been used in several areas of activity, due to its excellent characteristics and properties, such as biocompatibility, flexibility, optical transparency and chemical stability. In addition, PDMS modified with other materials promote desired changes to broaden its range of applications in various fields of science. However, the heating, mixing and degassing steps of the manufacturing process have not received attention in recent years when it comes to mixtures with solid materials. For example, PDMS has been extensively studied in combination with waxes, waxes that are usually in the solid state of matter at room temperature, making the interaction and manufacturing process difficult. Thus, we report the development of a multifunctional vacuum chamber in order to facilitate and accelerate the manufacturing process of PDMS composites combined with additives, blends and mainly solid materials. The multifunctional vacuum chamber developed in this work provides control of mixing speed parameters, temperature control and internal pressure, and can be used for other possible modifications with various materials and processes with approximate control of these parameters. As a result, samples manufactured using the multifunctional vacuum chamber achieved a time improvement of more than 133% in the heating and mixing step and approximately 200% in the last degassing step. In the complete manufacturing process, an improvement of more than 150% in preparation time was obtained. The multifunctional vacuum chamber can be used for other mixtures and manufacturing processes where it is desirable to control temperature, agitation speed and negative internal pressure in relation to atmospheric pressure.

**Keywords:** Manufacturing; PDMS composites; Vacuum chamber; Beeswax; Project; Polydimethylsiloxane (PDMS); Fabrication process; Gravity casting.

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<sup>1</sup>. Ariati, R. et al., 2021. Multifunctional low-cost vacuum chamber to manufacturing PDMS based composites. Article in submission process in Journal Machines, MDPI.

### 3.1 Introduction

Elastomers have become popularly known in several scientific areas in recent decades due to their excellent characteristics and properties, such as: flexibility, biocompatibility and corrosion resistance [1]. PDMS is, among the various existing elastomers one that has been receiving attention among researchers in various areas in recent years. PDMS is a mixture of linear polymers belonging to a group of silicones based on organic polymers. This material is non-toxic, non-flammable and has excellent mechanical, thermal and chemical properties [2–4]. PDMS has been applied in several areas, as the use in electronic, microfluidic and industrial devices has been reported [5–7]. The main characteristics of this material are its highly flexible [8], biocompatibility [9], optically transparent [10] and low cost. This makes material have a wide applicability and improvement of some aspects such as superhydrophobic surfaces, water repellent [11], anti-icing, self-cleaning [12] and anti-scaling [13].

Manufacturing processes commonly used with PDMS are numerous, depending on the mix and current needs, and can be complex processes or the use of expensive or unavailable equipment [14–16]. Some of these processes are the inlaying method, which consists of dissolving the PDMS that has been cured by TEOS under bis and DOTDL catalysis, and the wax weight ratios were followed by an ultrasonic treatment. Subsequently, a piece of immaculate tissue was dipped into the mixture, forming a layer of the mixture on the tissue surface, leaving it superhydrophobic [17]. Other processes are carried out with the cured PDMS immersed in a bath of paraffin resulting in the impregnation of the paraffin in the reticulated network of the PDMS. Another method also represents mixing uncured PDMS with paraffin / organic solvent and curing agent. And another way to mix is to put the PDMS in a liquid state with molten paraffin and then add the curing agent followed by mixing [18]. However, to create superhydrophobic coatings the spray method is used, which is composed of PDMS, THF, beeswax and curing agent, after spraying on the surface it is necessary to raise the temperature to evaporate the THF and cure the mixture [19]. The method trivially used in the composition of mixtures with waxes is gravity casting, which consists of mixing PDMS and waxes, paraffin or beeswax, followed by heating and mixing to impregnate the elastomeric matrix, after this step add the curing agent, mix and pour into molds. The last step consists of the degassing process [20]. These processes can also be used for PDMS combination with other materials. For instance, the literature presents works with

composites with the elastomer and additives, waxes, fibers, micro and nanoparticles [21–25]. However, the main problems pointed out are: As waxes are usually in their solid-state of matter at room temperature, mixing it with PDMS can cause some problems and require great attention.

Heating, as well as temperature control, are ideal for the perfect and efficient mixing to occur with the PDMS, which is in a liquid state, which is why the first heating step is so important[26–28]. In general, the mixtures should be agitated for a better distribution of the mixture and also more quickly. However, this agitation is often done manually due to the lack of equipment, simple or sophisticated, or even use some type of equipment for this step, however, it is a piece of high-cost equipment, not being convenient and available for all research[29–32]. And finally, it is time to remove the air bubbles from the previous stirring step, as these two steps are not usually carried out simultaneously, it takes longer to remove the bubbles from the mixture, as the surface tension of the PDMS is high and it is not let them out. In the conventional manufacturing method, many studies perform this step with approximately the same time of 30 minutes, taking into account that large-scale production is not the most efficient method[33–37].

The objective of this work is to design, develop and manufacture a multifunctional vacuum chamber (MVC) capable of improving the large-scale production process of PDMS being mixed with other materials. Many of the conventional processes reported in the literature do not have control of the parameters of each step of the process, with the occurrence of errors and defects caused by the operators handling the mixture being noticeable. Another detail is that the process does not have a pattern, thus causing non-repeatability of the process. The multifunctional vacuum chamber focuses on improving simultaneous steps, compounding quality and time reduction, making the entire process efficient for large-scale production.

### **3.2 Materials and methods**

For the manufacture of the multifunctional vacuum chamber, the following materials were used: 3 mm thick A36 steel sheet, 10 mm thick transparent acrylic sheet and the two-component poly (dimethylsiloxane) was selected, in which part A was selected. contains a prepolymer and part B is the catalyst to effect the cure.

The equipment used to control the motor rotation speed and the water bath temperature was a PWM 12v dc and STC-1000, respectively. The supports and covers

were manufactured in 3D printing. The vacuum pump used was a two-stage piston with a flow capacity of 143 l/min and a vacuum range of 13.53 psi. The vacuum chamber (2.1) stabilized when the pressure is down to 0.01 MPa [38]. A two-component poly(dimethylsiloxane) was selected, in which part A contains a prepolymer and part B is the catalyst to effect the cure. The beeswax used was purchased from a local (Bragança, Portugal) beekeeper.

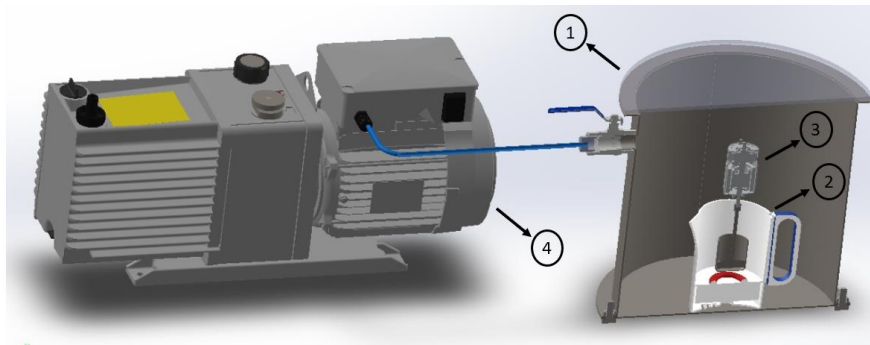
### 3.2.1 Design

The main mechanical objective for developing and manufacturing of this multifunctional vacuum chambers was to simplify and improve the efficiency of the process, as difficulties were reported during the mixing and degassing process, as well as the control of parameters during the manufacture of PDMS compounds[36]. The design of the multifunctional vacuum chamber was developed in the 3D CAD tool SolidWorks 2020, a license made available by the Polytechnic Institute of Bragança.

The developed multifunctional vacuum chamber is composed of an electric kettle and an electric perforation motor adapted inside the chamber to compose the heating and mixing steps of the composite, respectively. Vacuum pump and electronic controllers are listed on the outside. The electric kettle is used to heat the water bath using the joule effect to mix PDMS / wax at the melting temperature. The electric drill motor is used to carefully mix the liquid compound. The vacuum pump is used to partially remove the air from the chamber in order to reduce the internal pressure, in such a way, facilitating the exit of the gas bubbles formed during the mixing and pouring steps of the molds. Figure 3.1 illustrates the elements of the system constituted for the simultaneous and sequential steps.

The electrical system is essential for the correct functioning of the entire system, both for feeding the vacuum pump and for feeding the controllers. The voltage required to power the temperature controller and the PWM speed controller are 220V and 12V, respectively. The PWM speed controller works with voltage and current variation to control the rotations per minute of the mixture, this equipment is connected to a power supply, lowering the voltage from 220V AC to 12V DC, whichever is used, feeds the motor of adapted drilling. The STC-1000 temperature controller works as an on and off switch of the electric kettle, so that when the electric kettle is turned on its operating power is always 100%. The vacuum pump uses 220V voltage to work properly. Figure 3.2 shows the functioning of the electrical system of the global system.

The main electrical diagram was developed to automate the process, being controllable outside the multifunctional vacuum chamber. Parameters that can be instantly adjusted are internal water temperature, compound agitation and negative atmospheric pressure. Thus, this multifunctional vacuum chamber is ideal for the manufacturing process of polymers and composites that require any heating, controlled mixing, negative atmospheric pressure or combinations of these parameters [15].



**Figure 3.1- Multifunctional vacuum chamber project (1. Vacuum chamber; 2. Electric kettle; 3. Rotative motor and 4. Vacuum chamber).**

### **3.2.2 Multifunctional vacuum chamber manufacturing**

#### **3.2.2.1 Manufacturing of mechanical elements**

During the manufacturing process of the multifunctional vacuum chamber, several cutting processes were used, such as: hydraulic, laser and manual guillotine. On A36 steel plates with a thickness of 3mm, the shear cuts were performed using a hydraulic guillotine and manual cuts were necessary due to the complexity of the plates. The cuts by the mini 320 hydraulic guillotine were performed through the relative movement of a cutting punch against a die, in this case, the cut is simple with a square die, being the cylinder body of the chamber. The manual cuts, being the support rings of the upper and lower lid of the multifunctional vacuum chamber, were performed with the grinding wheel equipment with a cutting disc.

The laser cuts were performed on the acrylic sheet with the 2-AXIS Portlaser x252 equipment because it is easy, fast and accurate. This cut is more complex and with acrylic thickness of 10mm, the following cutting parameters were used: Speed 10.67mm/s, Power 77.6W and PPI 400. Due to the thick plate, it was difficult to cut and had to be executed several times. times for the laser can cut completely without damaging the plate.

In this stage of manufacturing the multifunctional vacuum chamber, a small handmade calender available in the laboratory was used. The device is composed of a set



of cylinders or rollers, with rotating movements and adjustable pressure. The use of this equipment was to bend the side of the MVC with the intention of forming a cylinder.

In this work, the MIG / MAG welding process was used to join the body of the multifunctional vacuum chamber to the support rings of the covers. The equipment used was the modular MIG 453 which uses the heat of an electrical arc continuously established between an electrode and the base metal to fuse the electrode tip and the base metal surface of the joint which is defined at 23.3V. The equipment was adjusted to weld the MVC body longitudinally and also to join the upper and lower ring together providing support for the acrylic cover and the lower part of the MVC.

Additive manufacturing technique that consists of developing the object graphically in computer software and subsequently manufactured by deposition of successive layers of material, which can be thermoplastics or other eutectic materials, on a heated platform. This process was used to fabricate the engine support with the metal cover and the support to connect the engine shaft to the mixing rod. The 3D printer used to manufacture the components is the Anet A8 Plus.

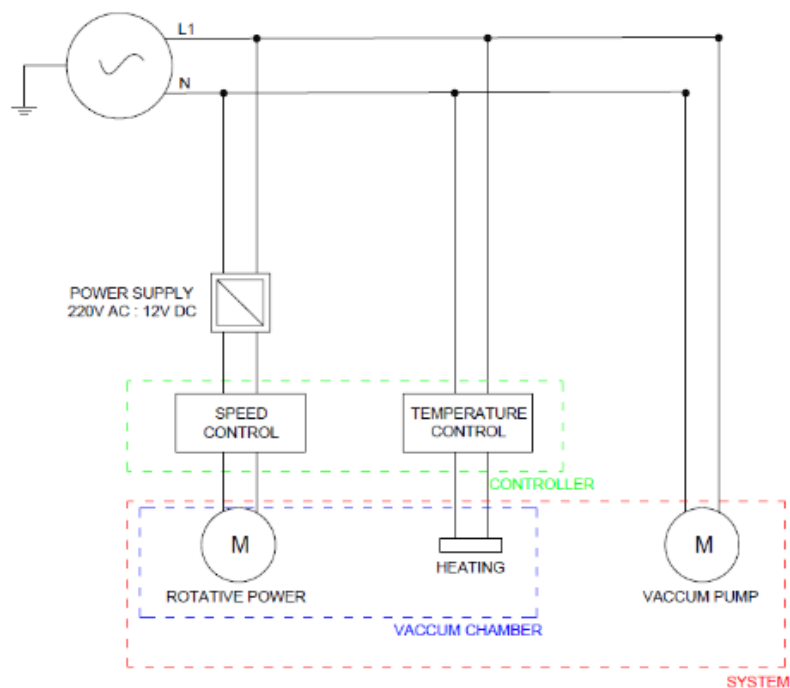


Figure 3.2- Operating electrical diagrams.

### 3.2.2.2 Assembly

The multifunctional vacuum chamber took shape in each process performed in the previous item, being a logical assembly sequence. First, the union of the support rings of the lower and upper covers with the cylindrical body of the MVC was made, as shown in figure 3.3a, followed by silicone above the weld for sealing. Then, the joint was made with screws and nuts for the cover and lower support with the aid of a silicone seal between them. To seal the acrylic lid and the upper support ring, a universal EPDM rubber adhesive tape was used between them. The parts manufactured in 3D printing consist of a box and a lid, both manufactured in PLA. The cover was fixed with support accessories also produced in PLA and by screws and nuts. The acrylic lid was cut again by the laser cutting equipment and then a 1/4" thread was made to fix the vacuum gauge that is in the center of the lid in order to measure the internal pressure.

The PWM speed controller is attached to the case made in 3D printing, is connected to a power supply which lowers the voltage to 12V DC and then power is supplied to the drilling motor positioned inside the MVC to make agitation. The STC-1000 temperature controller also located in the 3D-printed housing receives the voltage and transmits it directly to the electric kettle positioned inside the MVC. The vacuum pump is connected to a ball valve by a 1/4" gas hose making partial withdrawal of air from inside the multifunctional vacuum chamber. The complete system is shown in figure 3.3b.



(a)



(b)

**Figure 3.3- Fabrication and assembly of the Multifunctional vacuum chamber: (a) Welding the support rings on the MVC body and (b) Complete set of vacuum chamber and accessories.**

### 3.2.3 Testing the vacuum chamber and specimen manufacturing

After the manufacturing of the vacuum chamber, it was implemented some tests to evaluate the influence of the main operating parameters (heating, mixer and vacuum) in que quality of the composites and were manufactured tensile specimens using the vacuum chamber and by the conventional method.

The polydimethylsiloxane used to manufacture the specimens was Sylgard 184 and the beeswax was obtained from a local beekeeper (specification in table 3.1). For the manufacturing a two-component kit was required, one part being the polydimethylsiloxane prepolymer and the second part the crosslinking agent containing a mixture of vinyl- terminated polydimethylsiloxane and hydrogen. PDMS cure occurs when a catalyst is added under pure PDMS or already mixed with other substances through a reaction between the vinyl and hydrosilane groups[39].

**Table 3.1- Properties of the PDMS and Beeswax [41–45].**

<b>Property</b>	<b>PDMS</b>	<b>Beeswax</b>
Viscosity (base)	5100cP	0.1 cP ratio
Viscosity (mixed)	3500cP	-
Working time (pot life)	1.5 hours	-
Cure time at 25°C	48 hours	-
Density	0.98 g/cm <sup>3</sup>	0.95 g/cm <sup>3</sup>
Kinematic viscosity (80°C)	-	8.2 mPas.s
Thermal conductivity	0.27 W/m <sup>°K</sup>	0.20 W/m <sup>°K</sup>

#### 3.2.3.1 Testing the vacuum chamber

The temperature controller is connected to the electric kettle, heating the water contained inside the kettle through the joule effect. The STC-100 temperature controller equipment located outside the MVC is programmable, having 2 functions F1 and F2, where F1 is the specific temperature and F2 is the variation above and below this specific selected temperature. For testing liquids, the F1 value is 65°C and the F2 value is 0.1°C. The liquid content is 500ml for all liquids.

Some liquids were tested at this stage, looking for the smallest variation in the temperature range to increase the accuracy of constant heating during the melting and mixing process. The liquids are listed in Table 3.2 with their respective temperature

ranges, along with the heating time and the cooling time, with water saturated with sugar being more suitable.

**Table 3.2- Tests performed with different liquids checking the temperature range.**

<b>Liquids</b>	<b>Range (°C)</b>	<b>Heating time</b>	<b>Cold time</b>
Vegetable oil	65 - 85	1:31 minutes	21:46 minutes
Detergent	65 – 79.9	1:01 minutes	18:22 minutes
Water saturated sugar	65 – 69.7	00:39 seconds	5:51 minutes

After the heating step of the composite, agitation is necessary for the wax to impregnate the PDMS matrix. To test the two methods, two metallic composite cups containing 20g of PDMS and a proportion of 0.2g of beeswax each were weighed. The heating and mixing process was carried out under the same conditions and times, at approximately 65°C, but with temperature control available in MVC and in the conventional method there is no such control. The metallic cups are placed in water bath, however, the metallic cup in the MVC is placed inside the electric kettle containing water saturated with sugar and the conventional method uses a double boiler inside a container containing water. The times that were performed the tests are 1, 2, 5 and 7 minutes for the conventional method and 1, 2 and 3 minutes for the method performed in the vacuum chamber in order to analyze the efficiency of the methods. Another test performed in this step is when the curing agent is added after the heating and mixing step and again returned to heat and mix with the curing agent, but the heat accelerates the PDMS curing process.

For the last stage of degassing, some tests were carried out. The maximum working pressure of the vacuum pump with the respective time was verified and the efficiency of the insulation of the multifunctional vacuum chamber was tested. First, the MVC was closed with the acrylic lid containing the lower-than-atmospheric pressure gauge and the time for the system to reach maximum working capacity was verified. In order to test the insulation of the united parts of the MVC, a test was carried out allowing the internal pressure to reach the maximum working capacity, so the ball valve was closed, trapping the internal pressure and verifying how long it would maintain full pressure.

The main steps to manufacture the composite PDMS / beeswax samples are: The first step is the mixing of the PDMS base polymer with the beeswax respecting the proportionality in weight. In the second step, it is necessary to heat the composites and carefully mix the two materials, when the mixture is homogeneous, the curing agent is added. Again, it is necessary to mix the composite, in the case of MVC it is already

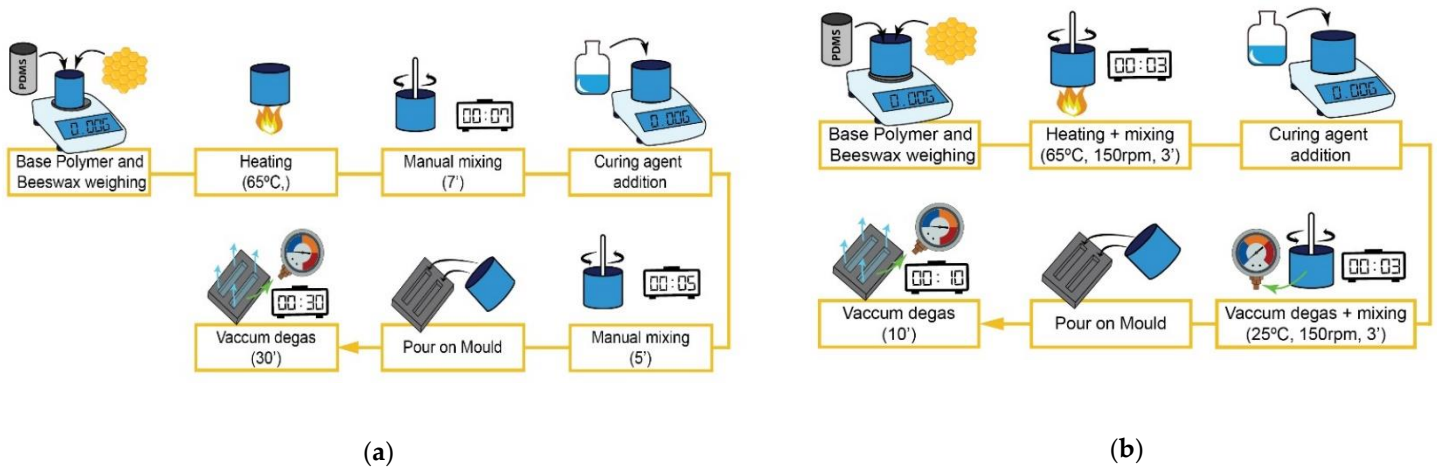
possible to do this step under vacuum action. Finally, the mixture is poured into the molds and degassed.

### **3.2.3.2 Conventional manufacturing method of specimens**

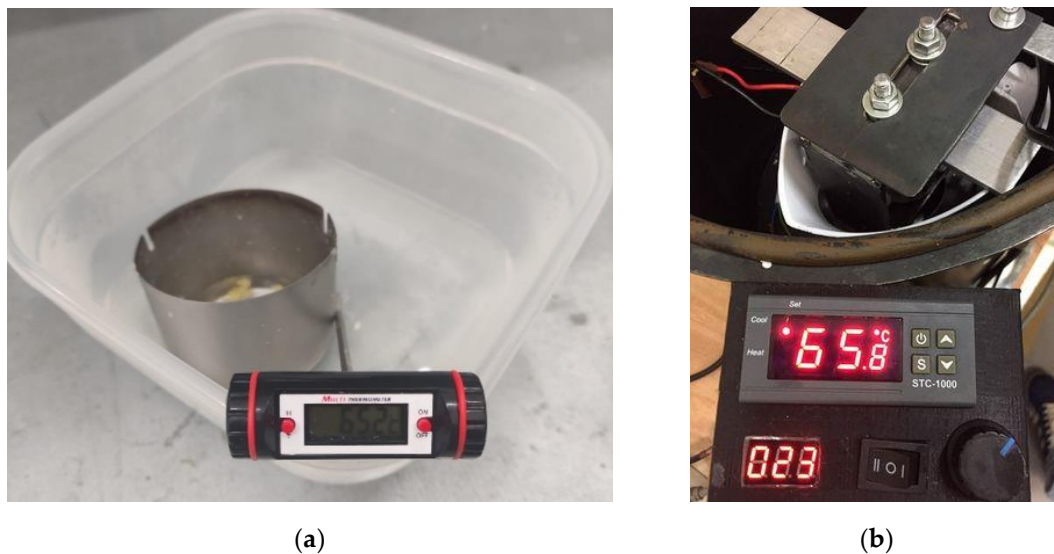
Samples were fabricated by combining PDMS with added beeswax (M\_BW1%). The preparation begins with weighing the PDMS and beeswax in the exact proportions for each mold, 5.5g of the polymer and 0.05g of wax were weighed. As PDMS and beeswax do not mix at room temperature, it is necessary to heat it to approximately 65°C as shown in figure 3.5a. The mixture was stirred with a metallic spatula, rotating clockwise for 7 minutes until the mixture was homogeneous. After this stirring process, the curing agent was added in a ratio of 10:1. Again, the stirring effect with the curing agent takes place for approximately 5 minutes and is then poured into the molds for testing. Finally, it was placed in a vacuum desiccator for 30 minutes to remove bubbles from the stirring and pouring process. The samples were left for 48h at room temperature of 25°C to complete the curing process. Figure 3.4a shows each step of the manufacturing process described above.

### **3.2.3.3 Specimens manufacturing using the developed vacuum chamber**

The manufacturing in the Multifunctional Vacuum Chamber holds control of some important parameters to improve the efficiency of the process, as the temperature and agitation speed. Also, it was able to do the degassing process while making agitation of the mixture inside the chamber. First, adding the beeswax (MVC\_BW1%) into the PDMS and weighing 5.5g of the polymer and 0.05g of beeswax as described in the previous process. The mixture was mixed using a mechanical stirrer with a speed controller, being stirred for 3 minutes with 23% of the maximum engine power until the mixture was homogeneous, controlling the temperature at approximately 65°C as shown in figure 3.5b. The second stage, the curing agent was added, followed by stirring in the MVC, making the process of instantaneous degassing for approximately 3 minutes and poured into the molds to carry out the tests. The last step in the manufacturing process is to remove the few bubbles from shaking and pouring into the molds at MVC for only 10 minutes. The samples were left for 48h at room temperature of 25°C to complete the curing process. Figure 3.4b shows each detailed step of the manufacturing process described above.



**Figure 3.4- Manufacturing process: (a) conventional manufacturing method of specimens and (b) Specimens manufacturing using multifunctional vacuum chamber.**



**Figure 3.5- Procedure for mixing the PDMS and beeswax: (a) Conventional method and (b) Method using Multifunctional vacuum chamber.**

### 3.3 Results and discussion

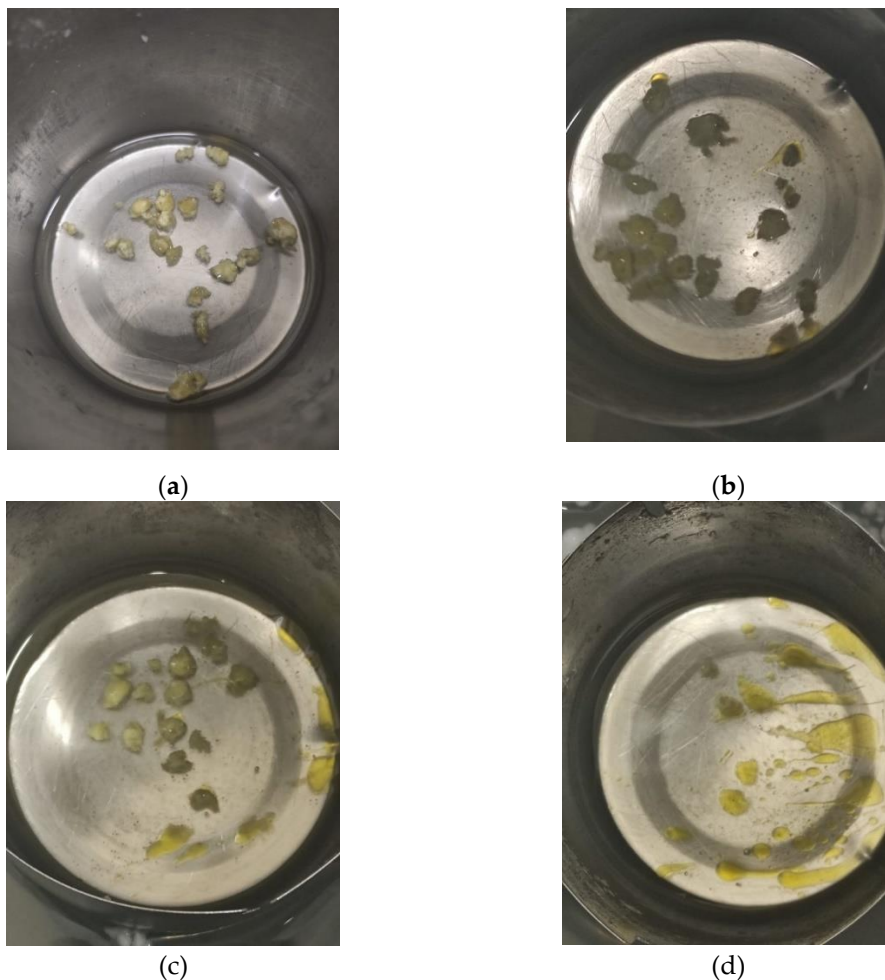
The test results for the process of mixing, heating and degassing the PDMS and beeswax composites are much more qualitative than quantitative, so the analyzes will be carried out to demonstrate the effects of each process step being controlled by temperature, time, agitation speed and pressure.

#### 3.3.1 Heating effect

The difficulty of this first stage of the process is temperature control, as the manufacturing processes described in the literature are carried out in a water bath without a temperature controller, thus losing process efficiency and negative results [15,29,46].

Temperature is the most important parameter for the homogeneity of the mixture, taking the beeswax to the liquid state of matter, facilitating and improving the interaction with the PDMS matrix. Figure 3.6 shows the action of temperature over time on the

beeswax contained in PDMS, which stays at a temperature of 65°C for approximately 10 minutes. This time depends on the amount of PDMS and beeswax that are directly proportional to time and temperature. As shown in Figure 3.6a, where it contains PDMS and unheated beeswax over time, where the beeswax does not dissolve in the PDMS matrix. However, in figure 3.6b it is observed that after 1 minute of heating, few signs of fusion begin to appear in the beeswax portions contained in the PDMS. In Figure 3.6c, one can more easily see the melting process of solid beeswax after 5 minutes of heating. Finally, in figure 3d, after 10 minutes of heating at approximately 65°C, the beeswax is completely in the liquid state of matter, making the interaction of beeswax in the PDMS matrix easier and more efficient.



**Figure 3.6- Effect of temperature on the PDMS and Beeswax mixture at 65°C over time: (a) No heating, (b) 1 minutes, (c) 4 minutes and (d) 10 minutes.**

### **3.3.2 Effect mixer**

After the essential heating process to improve the interaction of the mixture with the PDMS, the multifunctional vacuum chamber must be able to agitate the mixture so that it becomes homogeneous more quickly. Since these processes are carried out in

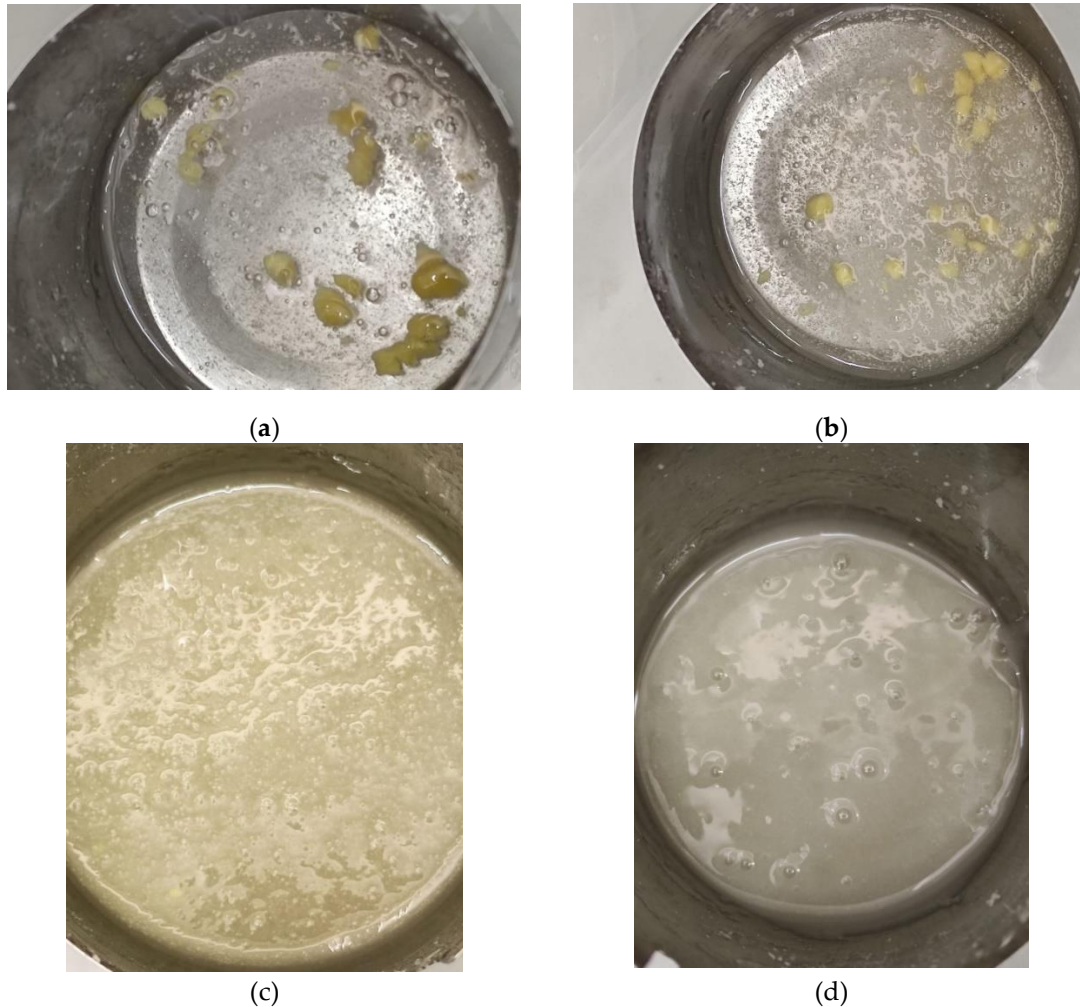
different stages that are not simultaneous due to the unavailability of equipment [32,47–49]. The difficulty of the process defines that the mixture is manually agitated without speed control and agitation uniformity, thus having the delay and low efficiency of the process. The multifunctional vacuum chamber has the possibility of working with heating control and mixing agitation control simultaneously and can be used for other experiments with numerous variations of the desired parameters.

Figures 3.7 and 3.8 show the comparison in illustrative images of the manual stirring process and the automated stirring process performed in the multifunctional vacuum chamber. In figure 3.7, manual agitation of the mixture composed of PDMS and beeswax was carried out along with constant heating at approximately 65°C. In figure 3.7a circular agitation was carried out with a metal spatula with rectangular section in a clockwise direction for 1 minute and show see that the wax is mostly in solid state. In the sequence of figure 3.7b the agitation continues in rotary motion for 2 minutes and we managed to repair a small decrease in the solid pieces of beeswax. Next image refers to the time of 4 minutes of constant clockwise stirring and show see a considerable decrease in the dissolved wax in the PDMS, but there are some small pieces of beeswax still in solid state as identified in figure 3.7c. The last illustrative image being the figure 3.7d completely in the liquid state and mixed with the PDMS leaving the homogeneous mixture in the equivalent time of 7 minutes.

The multifunctional vacuum chamber was developed with the aim of improving and speeding up the process steps for an effective and efficient series production. The mixing mixing process in the multifunctional vacuum chamber was automated with the potential to control the rotations for minutes provided with the PWM speed controller. Figure 3.8 shows the dissolution of beeswax in polydimethylsiloxane as a function of time by the automated mixing method. Figure 3.8a illustrates the blending of polydimethylsiloxane with beeswax starches in the solid state of matter without stirring and heating for comparison. Figure 3.8b shows the mixture made with 23% of the engine's maximum power for 1 minute and heating to approximately 65°C, identifying a rapid disaggregation of the beeswax in the PDMS. In the next illustrated image of figure 3.7c after 2 minutes of stirring, the considerable decomposition of the beeswax can be seen. In Figure 3.8d, the beeswax is completely dissolved in the polydimethylsiloxane matrix within 3 minutes of stirring. The comparison between Figures 3.7 and 3.8 shows that the use of this automated method causes a considerable reduction in the wax dissolution time



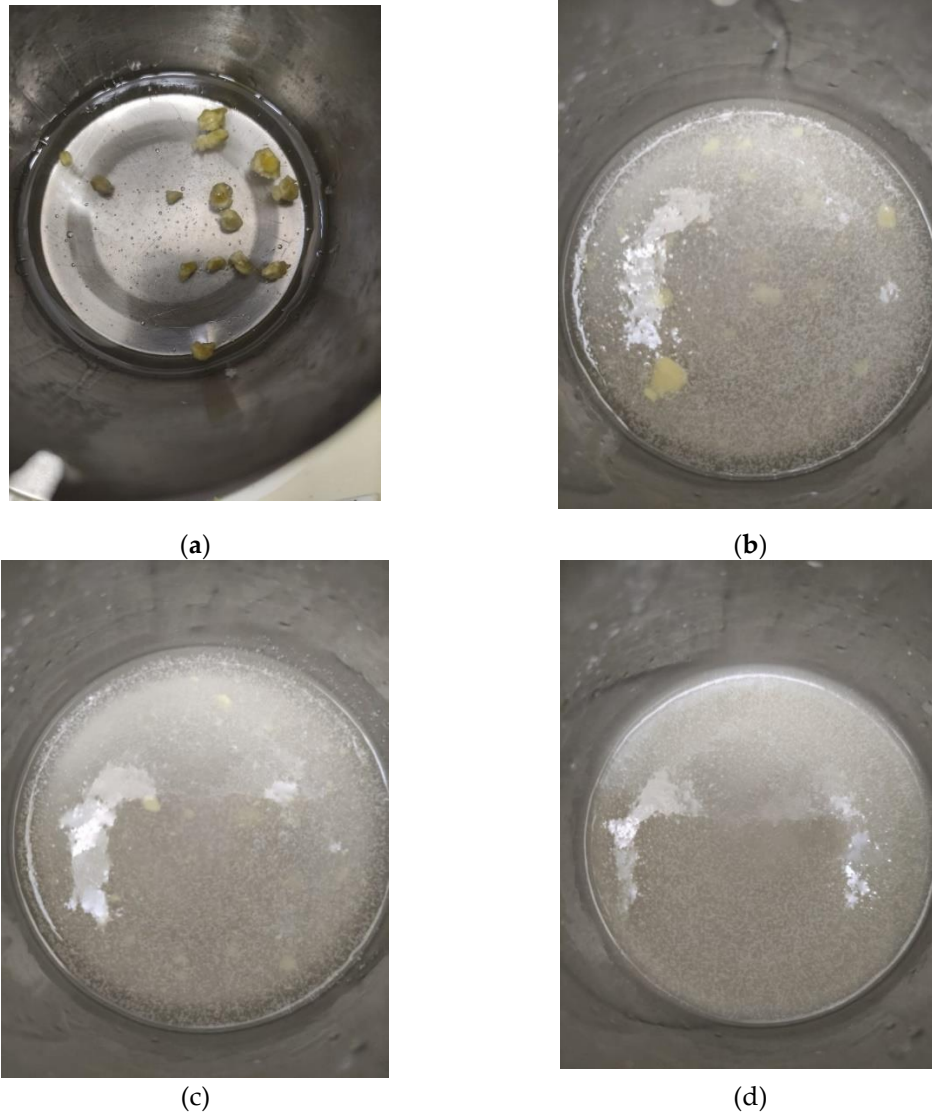
in the PDMS. Constant controlled agitation refers to a better distribution of the wax and, consequently, faster, as the heat from the water is evenly dissipated throughout the composite mixture. The agitator obtains a mixing paddle that occupies a larger area than the process is done manually with a metal spatula.



**Figure 3.7- Manual mixing of PDMS and Beeswax at a constant temperature of 65°C with respect to time: (a) No stirring, (b) 2 minutes stirring, (c) 5 minutes stirring and (d) 7 minutes.**

Compared to heating the composite without mixing and with mixing there is a considerable decrease in wax melting, however the time to completely melt the wax is 50% less.

In figures 3.7 and 3.8 it is possible to see a difference in color, texture between the two composites, there is also a significant decrease in the mixing time of the methods, with the conventional method taking 7 minutes and the method using the vacuum chamber multifunctional costs a time of 3 minutes. The composite mixed in the vacuum chamber with temperature and speed control improved by 133% in mixing time when compared to the conventional method.



**Figure 3.8- Automatic mixing of PDMS and Beeswax at a constant temperature of 65°C with respect to time: (a) No stirring, (b) 1 minutes stirring, (c) 2 minutes stirring and (d) 3 minutes.**

### **3.3.3 Effect vacuum**

Polydimethylsiloxane is present in works published in recent years due to its hydrophobicity, the fact that the molecular adhesion forces between water and the surface are weaker than the cohesion forces.

The high surface tension of polydimethylsiloxane creates a problem in the manufacture of specimens and does not allow the air bubbles generated in the stirring process to be removed, this fact happens at the interface between two chemical phases, making this surface layer of the liquid come to act as an elastic barrier. Atmospheric pressure makes pressure against the surface, making it difficult for the gases trapped inside the mixture to escape. In order to remove these inconvenient bubbles, a process called degassing is used. This process consists of applying negative pressure in relation to atmospheric pressure on the material in the liquid state, and by this pressure difference,

the dissolved gases of the liquid compound are eliminated. These bubbles generated due to agitation of the mixture cause defects that can later cause distortion in the tests, causing results different from the real one.

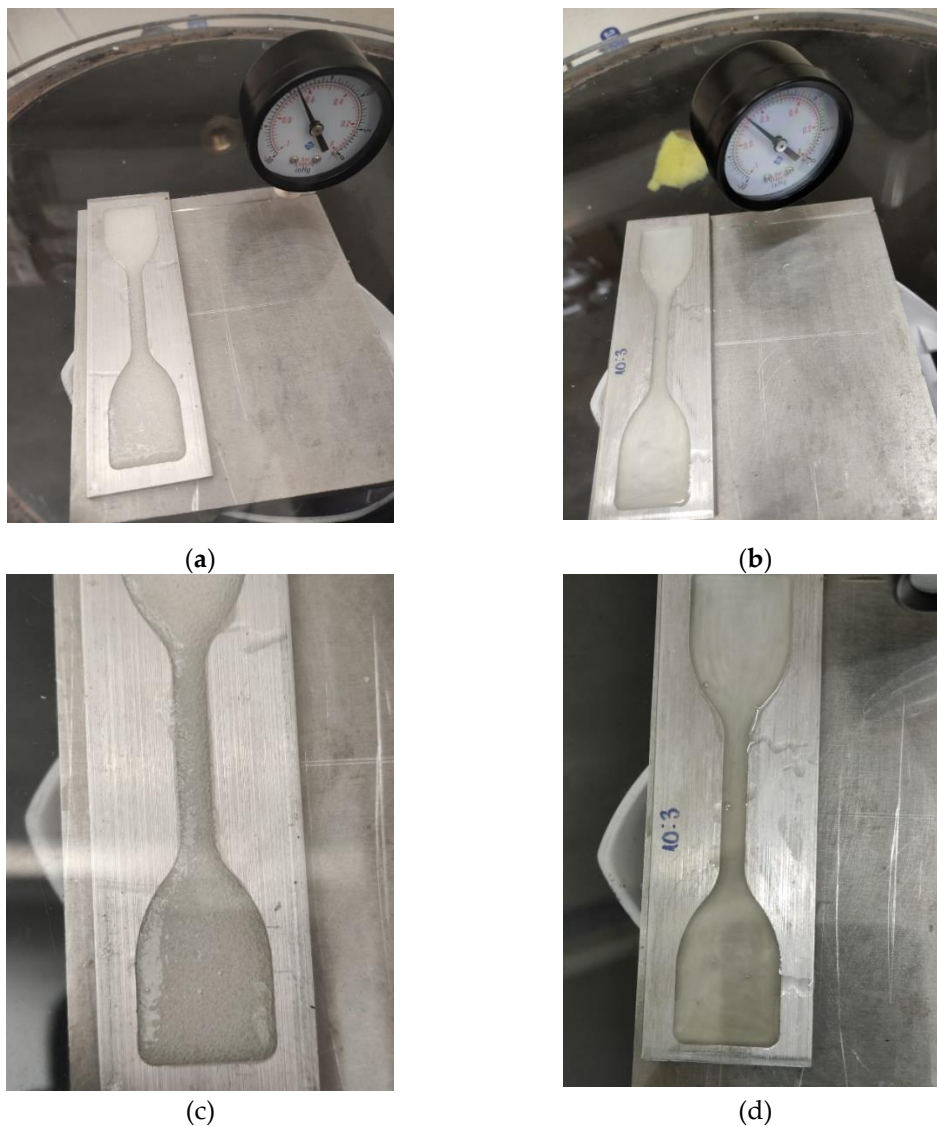
Figure 3.9a demonstrates the last step of the process prepared by the conventional method in a time of 1:10 minutes at a pressure of 13.4 psi in the vacuum gauge, with the existence of many bubbles on the upper surface of the mixture being visible, as shown in figure 3.9c. In addition, figure 3.9b shows the last step of the process developed by the new method employing the use of the multifunctional vacuum chamber in a time of 1:15 minutes at a pressure of 13.5 psi, with few bubbles being observed on the liquid surface as shown in the figure 3.9d.

It is possible to clearly observe the number of bubbles existing on the surface, as shown in figure 3.9c, produced by the conventional method due to the non-use of the vacuum action together with the final stages of the process to release the bubbles generated during the mixing step. However, in figure 3.9d there are few bubbles on the upper surface of the composite, which comes from the step in which the curing agent is added and mixed under the action of vacuum in the multifunctional vacuum chamber, thus carrying out the degassing step together with the curing agent mixing step resulting in reduced time. This new method, developed at MVC, takes approximately 3 minutes to degas, while other works reported in the literature are performed in a time equivalent to more than 40 minutes [35,37,46,50].

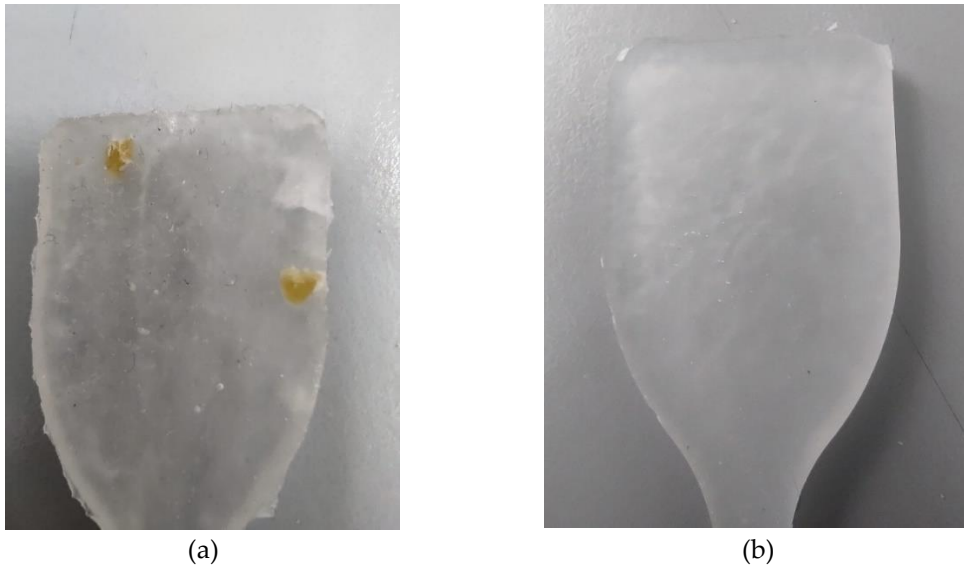
It is possible to clearly observe the number of bubbles existing on the surface, as shown in figure 3.9c, produced by the conventional method due to not using the vacuum action together with the final steps of the process in order to release the wetness generated during the mixing step. However, in figure 3.9d there are few bubbles on the upper surface of the composite, which comes from the step in which the curing agent is added and mixed under the action of vacuum in the multifunctional vacuum chamber, thus making the degassing step together with the step of mixing the curing agent resulting in the shortening of time.

One way to try to avoid and speed up the process without bubbles or even small pieces of wax would be to carry out all the steps under the action of a vacuum, but in the tests carried out in this work when it is heated and already mixed with the curing agent, this heat works as a cure accelerator as at 80°C the mixture cures completely in just 20

minutes, according to the manufacturer, and there is no time to finish the process because the mixture starts its curing process quickly, resulting in incomplete melting of the beeswax and bubbles trapped inside the samples. Figure 3.10a shows small pieces of beeswax that did not melt and some bubbles that were not removed because the mixture quickly solidified. Due to this relevant factor, this process was divided into several steps as described in the materials and methods so that the mixture did not cure before the wax was in the liquid state of matter and the bubbles were removed. Figure 3.10b shows a sample manufactured by different steps being heated and mixed, then added the curing agent and mixed at room temperature under vacuum action in the MVC and then poured into the molds under vacuum action again.



**Figure 3.9- Comparison between the methods of the degassing process. (a) conventional method in time 1:10 minutes and pressure 13.4 psi, (b) new method on MVC at time 1:10 minutes and pressure 13.4 psi, (c) conventional method sample and (d) new method sample.**

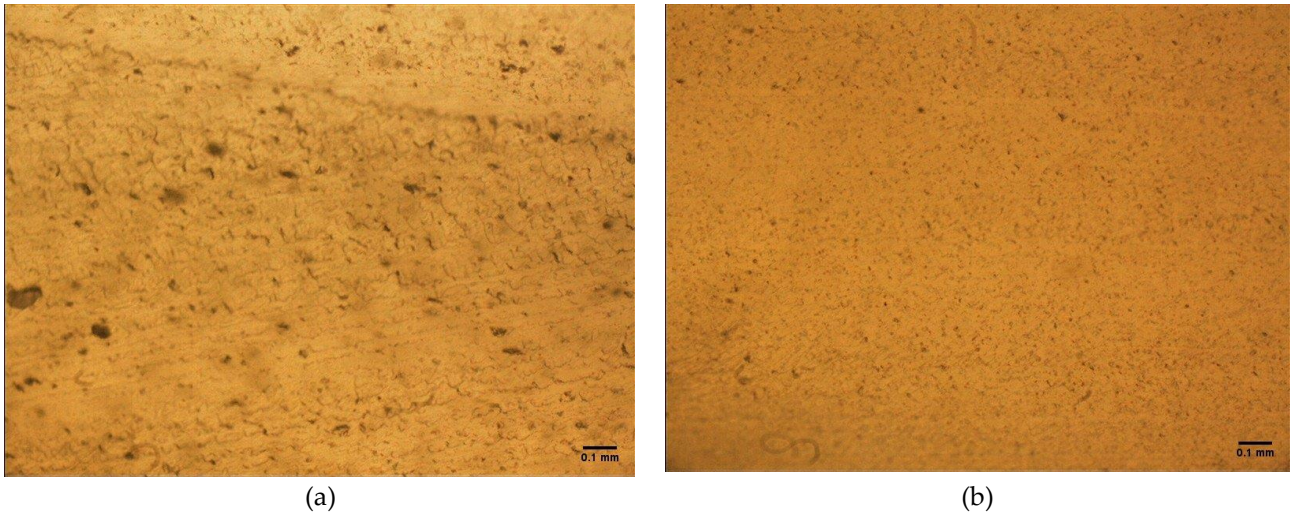


**Figure 3.10- Samples after curing: (a) Sample with curing agent performed with heating and (b) Sample with curing agent performed without heat and with vacuum.**

Figure 3.11 shows the uniformity of beeswax dispersion in the PDMS at a 100x scale. Figure 3.11a shows the sample performed by the conventional method and it is possible to notice that the wax particles are of non-uniform size. The sample produced by the multifunctional vacuum chamber has more uniformity in a certain region and smaller particles, as shown in Figure 3.11b, while the sample made by the conventional method shows disorder throughout the region and disorder in particle size. It is concluded from the electron microscope images that the samples manufactured using the multifunctional vacuum chamber show greater uniformity of beeswax in the PDMS.

### **3.4 Conclusion**

To improve the PDMS manufacturing process with different wax compositions that require heating, mixing and atmospheric pressure difference or even some of these simultaneous steps, this study was developed. Thus, the main objective is the development and production of a multifunctional vacuum chamber to assist in the manufacture of these composites that have these parameters in order to accelerate the manufacture of composites without losing their main characteristics. Generally, these samples are manufactured by a simplified conventional method without controlling the temperature, mixing and pressure parameters, so there is no repeatability and standard conditions for the process.



**Figure 3.11- Microscopic results: (a) Conventional method and (b) method using the multifunctional vacuum chamber.**

The multifunctional vacuum chamber played a very important role for the composites manufacturing area involving polymers, waxes and other additives. The MVC works for heating, mixing and negative pressure in relation to atmospheric pressure being programmable and controllable these parameters, also subject to being simultaneous or separate. Regarding the materials tested in this study, Poly (dimethylsiloxane) and beeswax, there was an improvement in all manufacturing steps. The main differences were the precision and the time, to perform the heating and mixing step the time decreased by more than 133% making this step faster. Going to the degassing step, an improvement of more than 200% was recorded compared to some works reported in the literature, due to the multifunctional vacuum chamber having the ability to perform two actions simultaneously, that is, mixing and vacuuming at the same time, making the step faster and more efficient. In general, the manufacture of samples using the multifunctional vacuum chamber proves to be faster and more efficient than when manufactured by the conventional method.

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## Chapter 4

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### Characterization properties of beeswax <sup>1</sup>

#### Abstract

Beeswax has been widely studied by researchers from various areas due to its characteristics of relatively fast decomposition and natural origin. Beeswax has excellent thermal and chemical properties. Beeswax has interesting characteristics of non-toxic, biodegradable. Its application is inserted in several areas, such as the food industry, cosmetics, pharmaceuticals and biomedicine. Among these areas, the main applications are in food packaging, skin protection products, medicines and waterproofing. However, beeswax changes composition and adulteration according to geographic location, climate, and other factors. Therefore, in this work a chemical test will be carried out to discover the origin and adulteration of this beeswax in question. The fourier-transform infrared spectroscopy (FT-IR) test was performed using a “compare” algorithm in the Spectrum software, this analytical technique produces a correlation between the spectra resulting in a factor equal to or greater than 0.98 sample is approved. The results obtained after using the software between the spectra of local beeswax and purified beeswax revealed a correlation equivalent to 0.996, meaning that this technique was approved and showing that there is no effective adulteration between the analyzed waxes.

**Keywords:** Beeswax, adulteration, Characterization, purified beeswax, “compare” algorithm, chemical properties

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<sup>1</sup> Ariati, R. et al., 2021. Characterization properties of Beeswax. Article in submission process in Journal of Composites Science, MDPI.

## 4.1 Introduction

Nowadays, environmental biology is being studied more and more by researchers from various areas due to the high load of waste generated throughout the planet. Scarcity of natural resources and global warming are issues that have been highlighted in recent decades. Biodegradable materials have received notable attention due to their short decomposition time and their sustainable character [1]. In this perspective, beeswax has been outstanding among materials of natural origin due to its excellent chemical and thermal properties [2,3]. Beeswax is synthesized from honey sugars and secreted in liquid form glands located in the abdomen, when it comes into contact with the atmosphere, it instantly solidifies. Beeswax has characteristics of non-toxic, biodegradable, ecological and sustainable [4–8].

There is a surprising rate of growth in studies involving beeswax in fact that its application is in several areas, for example, in the food, cosmetic, pharmaceutical and medicinal industries and others [9–15]. Among these areas, the main applications are in packaging for food preservation [16–18], medicines, burns, preservation of statues [19–22] and waterproofing [23]. These applications are justified by the beeswax retaining excellent properties such as biocompatible [24], extremely ductile, biodegradable [25], antibacterial [26,27] and humidity resistance [28].

Biodegradable products have been studied and tested to replace petroleum-derived products with the purpose of preserving nature, so beeswax has been tested and analyzed for uses in various sectors of food packaging. Oliveira et al produced biopolymer coatings to maintain the chemical and sensory characteristics of fruits and vegetables. Beeswax was added as a hydrophobic agent in the polymer network. Biofilm containing 10% beeswax showed the best results in water vapor transmission rate, 80% in elasticity and 15% decrease in solubility. The physicochemical analysis showed that it minimized weight loss, ensuring adequate fruit ripening for 15 days in a regulated environment. Thus, ideal coating for storing fruits and vegetables [10].

Beeswax has been extensively tested due to some characteristics that can modify the properties of composites, such as contact angle with water, permeability to water vapor, solubility in water. Cheng et al proposed to study several bloom films of gelatins (starch/gelatin-beeswax) testing their properties manufactured by extrusion blow. The samples showed an increase in the contact angle and lower permeability to water vapor

[29].

Wardhono et al proposed to develop an emulsion-based edible film based on a matrix of chitosan nanoparticles with CNCs. The oil-in-water emulsion was prepared by dispersing beeswax in an aqueous colloidal suspension of chitosan nanoparticles. There was an improvement in the water resistance properties and the mechanical strength reached 62.5 MPa with the addition of 25%wt of beeswax [21]. Omar-aziz et al proposed a study for PU edible films. Physical test results showed an increase in waterproofing properties synergistically, including water vapor permeability and water contact angle. PU-OSA films had poor mechanical properties, films containing beeswax had excellent properties in this regard [15].

The main objective of this study is to characterize the local beeswax in the northeast region of Portugal, the intention is to discover the purity of beeswax adulterated with microcrystalline wax, stearic acid, tallow, and paraffin by a modified Fourier-transform infrared spectra assay.

## **4.2 Materials and Methods**

The materials needed to carry out the tests were melted beeswax, stearic acid, calf, microcrystalline wax and beeswax comb (without adulteration). Beeswax comb and melted beeswax were purchased from local beekeepers (Bragança, Portugal). Beef cebo was purchased in a meat butcher (Bragança, Portugal). Stearic acid and microcrystalline wax were made available by the Polytechnic Institute of Bragança.

### **4.2.1 Fourier-transform infrared spectroscopy (F-TIR)**

FTIR spectra of the waxes were determined in an Universal Attenuated Total Reflectance (UATR) mode using a FTIR spectrometer Spectrum Two, Perkin Elmer, USA, in the 450 to 4000  $\text{cm}^{-1}$  zone, using 16 scans. Acquisition software used was Spectrum IR. No specific preparation was required for the analysis.

## **4.3 Experimental**

In this study Infrared (IR) was the analytical technique adopted for this purpose. Reference spectra of standard, in this case purified beeswax, was collected and saved for the sample as the raw materials known as beeswax adulterants and compared the spectrum of the incoming material against those reference standard spectra. The Spectrum software used the patented “Compare” algorithm for this purpose. A perfect match between the unknown and the reference spectra will yield a result of 1.00 with the Pass/Fail threshold

normally set to 0.98. Data has been collected using a Spectrum Two FT-IR Spectrometer equipped with a UATR (diamond ATR) sampling accessory. Spectra were measured at 4  $\text{cm}^{-1}$  of resolution.

## 4.4 Results and discussion

### 4.4.1 Fourier-transform infrared spectroscopy (F-TIR)

In Figure 4.1 the spectra of the beeswax sample acquired at is shown with the respective peak table. Figure 4.2 shows the spectra for the purified beeswax sample and its usual adulterants.

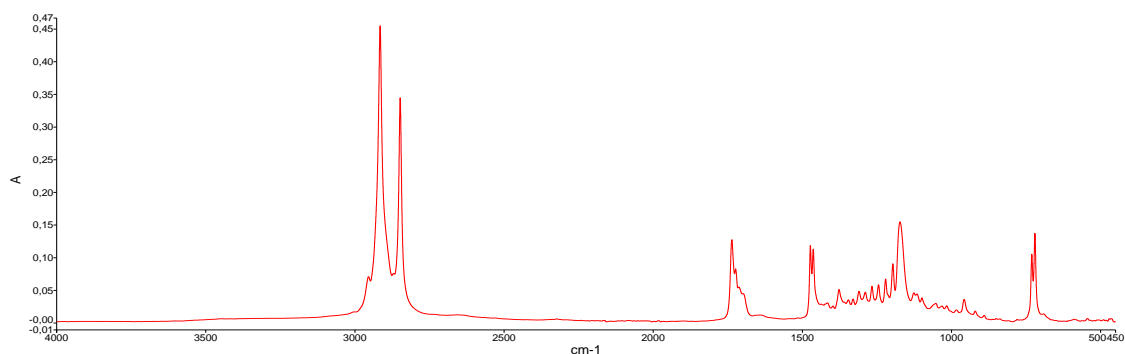


Figure 4.1- The spectra of the beeswax sample acquired.

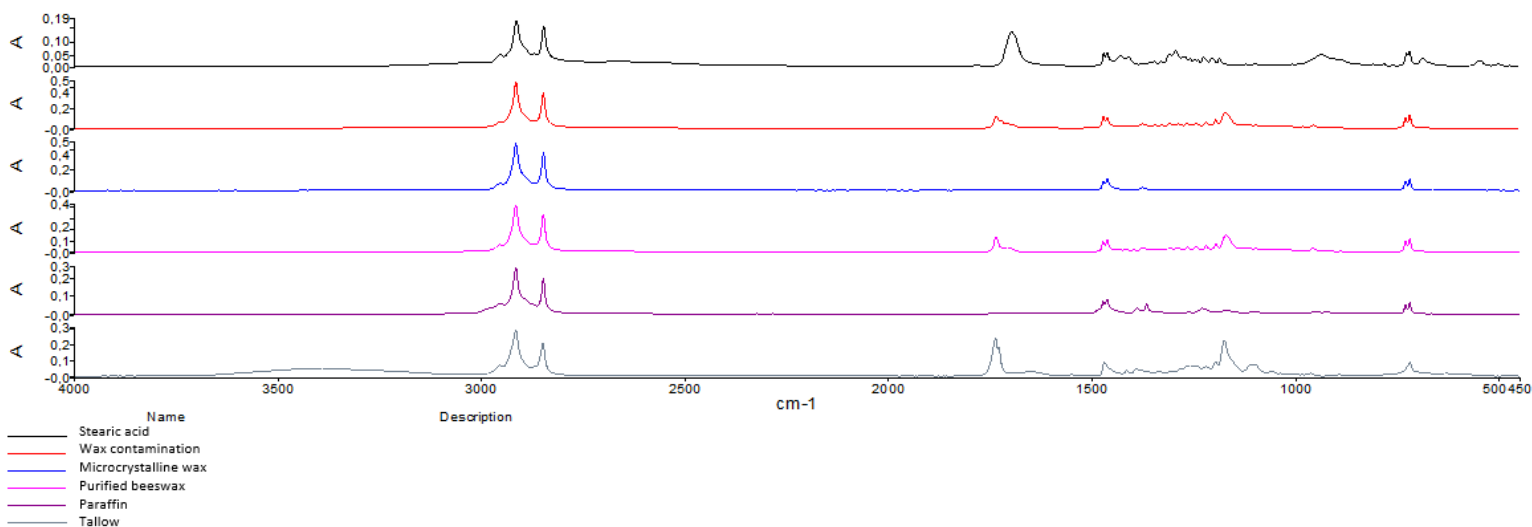


Figure 4.2- spectra for the purified beeswax sample and its usual adulterants.

Beeswax is a complex chemical mixture of monoesters; diesters; triesters; hydroxy monoesters; hydroxy polyesters; acid esters; acid polyesters with a total of 67%; hydrocarbons in 14% ; free acids in 12%; alcohols in 1% and others in 6% [33].

In the Figure 4.1 we can observe the absorption bands related to alkanes can be assigned: C–H stretching (at 2916 and 2849  $\text{cm}^{-1}$ ). The vibration at 2957  $\text{cm}^{-1}$  (shoulder) is assigned to the asymmetric stretching band of a  $\text{CH}_3$  group. The two intense bands at

2916  $\text{cm}^{-1}$  and 2849  $\text{cm}^{-1}$  are due to  $\text{CH}_2$  asymmetric and symmetric stretching vibrations, respectively [34]. The band observed at 1463  $\text{cm}^{-1}$  is assigned to  $\text{CH}_2$  scissor deformation or bending vibrations. At 1172  $\text{cm}^{-1}$  a band is observed and is attributed to stretching and C–H bending vibrations and the  $\text{CH}_2$  rocking mode methyl, seen only in long-chain alkanes is assigned at (719  $\text{cm}^{-1}$ ). Spectral region between 1800 and 800  $\text{cm}^{-1}$  has been considered as a fingerprint region for Beeswax because of a high number of absorption bands. The sharp band between 1700 and 1750  $\text{cm}^{-1}$  in our sample at 1736  $\text{cm}^{-1}$  occur from the carbonyls in the ester linkages between the fatty acids and glycerol backbone. This absorption can be attributed to the monoesters present in beeswax, which are the major ester component (>60%) of beeswax. Band 1376  $\text{cm}^{-1}$  was stated as a characteristic band in Beeswax [16].

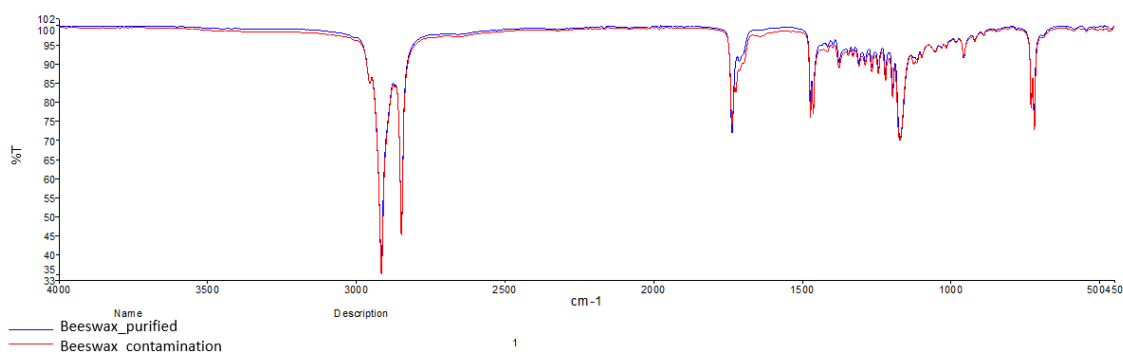
Figure 4.2 shows the ATR–FTIR spectrum of the most common beeswax adulterants, i.e., paraffin, microcrystalline wax, tallow and stearic acid. Obvious differences between the infrared spectra of beeswax and those of the adulterants can be observed. For paraffin and microcrystalline wax, as expected, no band is observed in the 1750–1700  $\text{cm}^{-1}$  range. The main bands for these two compounds are the vibrations at 2955  $\text{cm}^{-1}$  (asymmetric stretching band of a  $\text{CH}_3$  group), 2916  $\text{cm}^{-1}$  and 2848  $\text{cm}^{-1}$ , which are due to  $\text{CH}_2$  asymmetric and symmetric stretching vibrations, respectively. Paraffin and microcrystalline wax exhibited similar spectra (Fig. 2), with the only noticeable difference being the intensities of the peaks at 2955  $\text{cm}^{-1}$ . Microcrystalline wax contains a higher percentage of ramified hydrocarbons comparing to paraffin that is composed of linear hydrocarbons, that is the reason of their variance.

For tallow, the relative intensity of the band at 1737  $\text{cm}^{-1}$ , which is attributed to the stretching vibration of the carboxylic groups involved in the ester bonds of the triglycerides, is significantly greater (A of 0.25) than that observed for beeswax (A of 0.13). Additionally, for stearic acid, the intensity of the band at 1700  $\text{cm}^{-1}$ , attributed to the stretching vibration of the carboxylic groups of stearic acid, is equivalent than that observed for beeswax [32].

#### 4.4.2 Correlation results

The result shown in Figure 4.3 is for the test sample of Beeswax where the sample passes the test with a correlation value greater than the Pass/Fail threshold of 0.98.





**Figure 4.3- “Compare” result between sample and purified Beeswax (reference).**

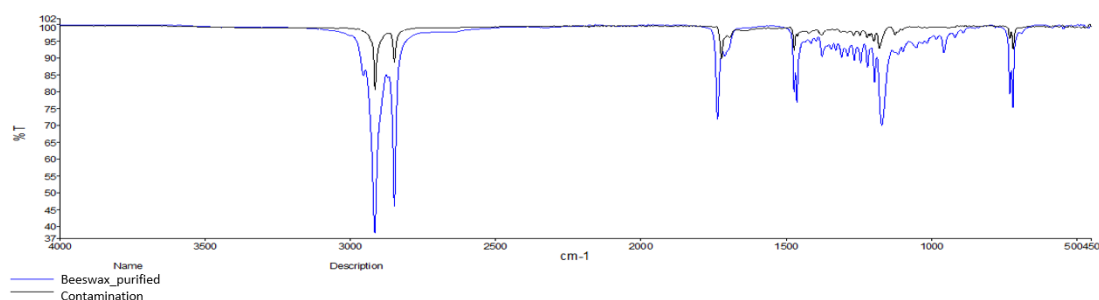
**Table 4.1 – Results of the infrared spectrum “compare”.**

Reference	Better result	Correlation	Pass / Fail
Beeswax_purified	Beeswax_contamination	0.996413	Pass

The infrared spectrum and the “Compare” results give us the information where gross contamination or adulteration has occurred. In cases where a material fails the “Compare” test, the material would normally be subjected to further testing by additional instrumental techniques or wet chemistry. In this study, we verified that our beeswax sample correlate to the purified beeswax in a 0.996413 value, as shown in table 4.1.

For further information, the difference between beeswax’s purified (reference) spectra and studied beeswax sample’s spectra was obtained. The resulting spectra belongs to the beeswax sample potential adulterant.

Sample of potential adulterant, returns the result shown in Figure 4.4 where the material “Fail” the “Compare” algorithm against the purified Beeswax (reference), the value is shown in table 4.2 confirming the result compared to the contaminated beeswax



**Figure 4.4- “Compare” result between Beeswax contamination and purified Beeswax (reference).**

**Table 4.2- Results of the infrared spectrum “compare”.**

<b>Sample name</b>	<b>Correlation</b>	<b>Pass/Fail</b>
Contamination	0.879437	Fail

Studied usual beeswax’s adulterants spectra were also compared with the one from Beeswax contamination, to check if any correlates with it. And the result is presented on table 4.3.

**Table 4.3- “Compare” result between usual beeswax adulterants and Beeswax (contamination).**

<b>Sample name</b>	<b>Correlation</b>	<b>Pass/ Fail</b>
Paraffin	0.882198	Fail
Stearic acid	0.874858	Fail
Microcrystalline wax	0.867876	Fail
Tallow	0.838976	Fail

#### **4.5 Conclusion**

The main objective of this study was to characterize the chemical composition of beeswax from the local region (Bragança, Portugal). However, to determine beeswax adulteration, the analytical Fourier-transform infrared spectroscopy technique was used for this purpose. After using the patented “compare” algorithm in the Spectrum software, a result of 0.996 was obtained in direct comparison with the beeswax used in this work for purified beeswax. Thus, it is possible to conclude that the wax used in this study has no adulteration and is approved by the analysis of the “compare” technique. An analysis was carried out using the same technique to compare the spectra that were not approved between the used beeswax and the purified beeswax matched the spectra of the adulterants, and when they were compared none was approved. Therefore, it is possible to conclude that the beeswax used is not adulterated by any of these adulterants and this difference between the spectra of the used beeswax and the purified beeswax may be due to the degradation of the beeswax.

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## Chapter 5

# Study of mechanical, optical and wettability properties of PDMS/Beeswax composite <sup>1</sup>

### Abstract

Polydimethylsiloxane (PDMS) is an elastomer that has received primary attention from researchers due to its excellent physical, chemical and thermal properties, together with biocompatibility and high flexibility properties. Another material that has been receiving attention is beeswax because it is a natural raw material, extremely ductile and biodegradable with peculiar hydrophobic properties. These materials are applied in hydrophobic coatings, clear films for foods and films with controllable transparency. However, there is no study with a wide range of mechanical, optical and wettability tests, and with various proportions of beeswax reported to date. Thus, we report an experimental study of these properties of pure PDMS with the addition of beeswax and manufactured in a multifunctional vacuum chamber. In this study, we report in a tensile test a 37% increase in deformation of a sample containing 1% beeswax (BW1%) when compared to pure PDMS (BW0%). The Shore A hardness test revealed a 27% increase in the BW8% sample compared to BW0%. In the optical test, the samples were subjected to a temperature of 80 °C and the BW1% sample increased 30% in transmittance when compared to room temperature making it as transparent as BW0% in the visible region. The thermogravimetric analysis showed thermal stability of the BW8% composite up to a temperature of 200 °C. The dynamic mechanical analysis test revealed a 100% increase in the storage modulus of the BW8% composite. Finally, in the wettability test, the composite BW8% presented a contact angle with water of 145 °. As a result of this wide range of tests, it is possible to increase the hydrophobic properties of PDMS with beeswax and the composite has great potential for application in smart devices, food packaging films, films with controllable transparency, water-repellent surfaces and anti-corrosive coatings.

**Keywords:** Polydimethylsiloxane (PDMS), Beeswax, mechanical and optical properties

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<sup>1</sup> Ariati, R. et al., 2021. Study mechanical, optical properties and wettability of PDMS/Beeswax composite. Article in submission process in Journal of Materials Science, Springer.

## 5.1 Introduction

Elastomers have received primary attention from researchers from different areas of activity, due to their excellent properties of elastic behavior and recovery of initial dimensions. Among elastomers, poly(dimethylsiloxane) has featured in a wide range of silicones. Another very important global factor that has brought satisfactory results for the environment is the use of biodegradable products. Among the biodegradable materials, beeswax has stood out among materials of natural origin. The use of these materials is due to their excellent physical, chemical and thermal properties [1]–[7]. Polydimethylsiloxane, more commonly called PDMS is a silicone rubber belonging to organosilicon polymers, while beeswax comes from a metabolic process of bees through the abdominal segments. Both PDMS and beeswax retain characteristics of non-toxic, biodegradable and also because they are materials whose processing is simple [8]–[13]. Another notable impact factor present in the use of beeswax is that it is a natural and sustainable raw material [14], [15].

There is a growing interest in studies of these materials due to the wide range of applications in different areas. Both PDMS and beeswax are evident in applications in the food, cosmetic, pharmaceutical and medicinal industry [16]–[24]. In the case of PDMS, the range extends to the areas of mechanical and civil engineering solutions, electronic devices and the biomedical field [25]–[29]. Among these areas, the applications for beeswax were diverse, such as food packaging [30], [31], burns, medicines, conservation of statues [32], [33] and waterproofing [34]. Polydimethylsiloxane is also broadly followed by filtration membranes [35], [36], microfluidic devices [37], [38], sensors, blood analogues [39], lubricants [40] and optical devices [41]. This wide range of applications is justified by the admirable properties exhibited by these materials. PDMS is a biocompatible silicone [42], [43], viscoelastic, chemically and thermally stable [44]–[46], highly flexible [47], corrosion and abrasion resistance [48], [49] and optically transparent [50]. However, beeswax has some similar characteristics such as being biocompatible [51], extremely ductile, biodegradable [52], antibacterial [53], [54] and humidity resistance [55].

PDMS and beeswax exhibit hydrophobic characteristics and are widely applied in coatings, resulting in surface enhancement with low surface energy [56]–[58]. The improved properties that can stand out are water repellent [59], anti-fogging [60], self-cleaning [61], oxygen permeability [62] and water vapor permeance [30]. Syafiq et al

presented a study to develop a transparent hydrophobic hybrid coating by adding PDMS to APTES. The hydrophobic coating showed improvement in self-cleaning, anti-fogging and transparency properties due to the presence of PDMS. After prolonged outdoor exposure at a 20° angle, the coating exhibited 84% transparency indicating excellent self-cleaning properties, the hybrid coating also exhibited anti-fogging behavior after prolonged exposure to mist and small water droplets disappearing after 6 minutes, consequently of these properties, the hybrid coating has high relevance for application on external surfaces of photovoltaic panels [59].

The development of materials with special wettability has been the main object of research for oil/water separation, and PDMS has favorable characteristics for this topic due to its hydrophobicity. Bolvardi et al developed a superhydrophobic coating based on titanium dioxide/PDMS nanoparticles and applied to different metal meshes with different pore sizes. Morphological analysis revealed that the presence of PDMS as a binder significantly altered the surface structure. The wettability results showed a superhydrophobic character and, in a mechanical durability experiment, PDMS demonstrated the role of improving the abrasion resistance [48]. However, in another study wang et al also presented a superhydrophobic surface and improved the abrasion and corrosion resistance for a modified cement mortar mixture with PDMS. Furthermore, another property that improved was tenacity and durability. The modified PDMS cement mortar can be used in concrete materials with impermeability and anti-corrosive characteristics, such as concrete pavement that uses de-icing salts [88].

Biodegradable products have been studied and tested to replace non-biodegradable products with the worldwide intention of preserving the environment, so beeswax has been bringing good results in food storage products. Reis et al proposed to produce a coating for biodegradable trays by immersion in beeswax emulsion in order to improve the barrier properties. The trays coated with 1% beeswax resulted in improved tensile strength and lower water solubilization capacity. The coating showed a reduction in water vapor permeability, making it ideal for the correct storage of fresh fruits and vegetables due to its adequate mechanical properties [15]. Bahrami et al developed a new AgNPs biocomposite ternary film for application in food packaging. There was an improvement in water vapor permeance, increased elongation at rupture and showed strong antibacterial activity [54].



The objective of this study is evidenced with the preparation of PDMS composite containing different proportions by weight of beeswax, with a focus on improving the hydrophobic properties of PDMS. So far there are no reports of studies mixing only PDMS and beeswax in various weight ratios, but the work contains a high amount of mechanical, optical and wettability tests followed by analysis and evaluation of mechanical, chemical, optical and wettability. This compound has great potential for application in many everyday products such as food packaging, controllable transparent films and transparent protective films.

## 5.2 Materials and methods

### 5.2.1 Materials

PDMS used to manufacture the samples was silicon-based silicone (Dow Corning Sylgard 184), consisting of a 2-component kit, part A being the polydimethylsiloxane prepolymer and part B being the crosslinking agent. The beeswax used was purchased from a local beekeeper (Portugal, Bragança). Specification of the PDMS in table 5.1.

**Table 5.1- Properties of the beeswax and PDMS.**

<b>Properties</b>	<b>PDMS</b>	<b>Beeswax</b>	<b>References</b>
Melting temperature (°C)	-49	60 - 65 65 – 70	[63],[6],[64] [30], [65]
Density 25°C (g/cm <sup>3</sup> )	0.98	0.95	[30], [66]
Kinematic Viscosity (mPa.s) 100°C		0.47	[30]
Viscosity	5100cP	0.1cP ratio	[6], [67]
Work time (pot life)	1.5 hours	-	[67]
Cure time (25°C)	48 hours		[67]
Thermal conductivity (W/m <sup>2</sup> K)	0.27	0.16	[66], [67]

### 5.2.2 Preparation of the specimens

The samples were manufactured by a special design equipment, a Multifunctional Vacuum Chamber that controls some important parameters to improve process efficiency, such as temperature, agitation speed and internal pressure. The complete fabrication of samples consists on 3 steps. First, beeswax was added to the PDMS and weighing 5.5 g of the polymer and 0.05 g of beeswax. The mixture was mixed in a mechanical stirrer with speed controller, being mixed for 3 minutes with 23% of the maximum engine power until the mixture was homogeneous, controlling the temperature at approximately 65 ° C. In the second step, the curing agent was added with the ratio recommended by the manufacturer 10:1 ratio, followed by the stirring in the chamber at multifunctional vacuum at a speed of 150 RPM, carrying out the process of simultaneous degassing for approximately 3 minutes and poured into metallic molds. The last step of the

manufacturing process is to remove the few bubbles from the previous step, then placed in the multifunctional vacuum chamber to remove the bubbles for just 5 minutes. Samples were left for 48h at room temperature of 25°C to complete the curing process. The samples were enumerated according to the amount by weight of beeswax. The samples identification were: 1<sup>st</sup> and 2<sup>nd</sup> characters (BW) indicate beeswax and the last characters refers the amount of wax in the group. For instance, BW0% means that the sample is made with pure PDMS, BW1%, indicates 1% of wax. In this work, tests were done at: BW0%, BW1%, BW2%, BW4% e BW8%.

### 5.2.3 Dynamic Mechanical Analysis (DMA)

Dynamic mechanical measurements were performed using the DMA Q800 V21.2 (TA Instruments). Two samples were made for each proportion of beeswax according to ASTM D7028 [68], with the following dimensions of length, width and thickness equivalent to 27 x 6 x 2 mm, respectively. The elastic (storage modulus) and viscous (loss modulus) components were measured while heating the samples at a rate of 3°C/min in the temperature range between room temperature and 130°C. The oscillation frequency was fixed at 1 Hz and the applied strain was 0.5%.

### 5.2.4 Thermogravimetric Analysis (TGA)

Measurements were performed using the SDT 2960 simultaneous DSC-TGA under inert material argon and air, heating the samples from room temperature to 400 °C. Two samples were used from the tensile samples of each percentage of 0% and 8% to perform the test and heated under the temperature program of 10 °C / min. The respective weights are listed in table 5.2.

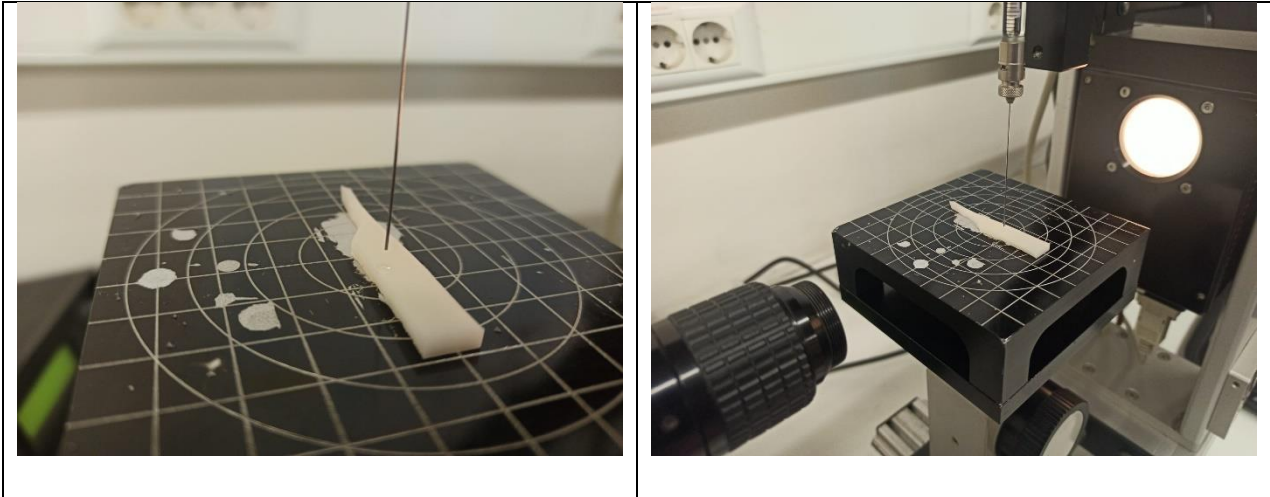
**Table 5.2- Mass of samples used in the TGA.**

<b>Specimen</b>	<b>Mass (mg)</b>
BW0% - Air	10.203
BW0% - Argon	11.523
BW8% - Air	10.985
BW8% - Argon	12.984

### 5.2.5 Wettability

The wettability test was performed in a Contact Angle System OCA equipment from Physics and the test was processed by the static sessile drop method, using distilled water as solvent. Two samples from the tensile samples were used and carried out at room temperature of, approximately, 25 ° C. The samples were placed on the surface of the equipment and the parameters of focus, luminosity and droplet deposition were adjusted

for each sample. The drop of distilled water was placed on the surface of the sample, as illustrated in figure 5.1, being intercalated every 5mm and the measurement was performed 5 times.



**Figure 5.1- wettability test: (a) positioning for dripping and (b) performing the test.**

### **5.2.6 Spectrophotometric test**

To perform the test, a spectrophotometer, model Shimadzu UV-2600 brand was used. One sample of each beeswax proportion group was used, samples from the tensile samples. The wavelength range created for the equipment was between 200  $\mu\text{m}$  and 800  $\mu\text{m}$ . Transmittance measurements were performed with the samples heated due to beeswax in the composition. To warm up the samples, a baby bottle heater was used in a bain-marie with the samples immersed in the water, raising the temperature to 80  $^{\circ}\text{C}$ . After a few minutes of heating, the samples were removed from the water, dried and placed on the spectrophotometer.

### **5.2.7 Tensile test**

Uniaxial tensile tests were performed with 5 specimens for each ratio in a universal testing machine, brand SHIMADZU, controlled by the software version 1.5.1. The tests were performed according to ASTM D412 [69]. To perform the tests, a pre-test was set up on the machine, with a speed of 5 mm / min being adjusted until it reached a preload of 1 Newton, and from this point, the test was set up for a speed of 500 mm / min until the sample breaks. To prevent slipping of samples during the test, fine sandpaper was placed on the ends of the samples.

### **5.2.8 Hardness test**

The test was performed in a portable Shore A analogue hardness tester according to ASTM D2240 [79]. To measure hardness, two specimens from each sample were

placed on a flat surface with the flatter side of the samples being chosen for testing. Five-point measurements were taken at an ambient temperature of approximately 25°C.

## **5.3 Results and discussion**

### **5.3.1 Dynamic mechanical analysis test**

In order to investigate the mechanical properties of composites analyzing their behavior with increasing temperature, the DMA test was performed on the manufactured samples. Figure 5.2 shows the behavior and the data were listed in Table 5.3. Figure 5.2a shows the graph of storage modulus versus temperature containing the superposition of the curves for pure PDMS (BW0%) and composites with 1% by weight (BW1%), 2% by weight (BW2%), 4% by weight (BW4%) and 8% by weight (BW8%) of beeswax. Composites made from PDMS and beeswax are amorphous, as can be seen. There was an increase in the storage modulus of composite BW1% when compared to pure PDMS, the values were approximately 1.7MPa to 3.1MPa, respectively. Interaction between PDMS and beeswax particles is more intense with increasing beeswax content. However, there was an unexpected behavior with the BW8% sample, slightly stepping back the storage modulus. The glass transition region of the composites formed are between 75 and 77°C. For the composite BW4% obtained a maximum storage modulus equivalent to 3.7MPa at approximately 30°C. The glass transition occurred close to 76.5°C entering the elasticity state at about 87°C and obtained storage modulus equivalent to 2.5MPa after the glass transition.

The BW8% reached the maximum storage modulus of 3.3MPa before the glass transition. The glass transition occurred at approximately 76°C and the elastic state started at around 87°C followed by storage modulus equivalent to 1.3MPa. When beeswax exceeds the melting point, particles move from solid to liquid in a uniform way, reducing interaction of beeswax with PDMS and justifying the decrease in storage modulus.

BW4% composite has the highest storage modulus than BW8% composite, but BW8% composite has the highest loss modulus before glass transition and lower T<sub>g</sub> compared to BW4% composite. The increase in loss modulus may be due to soft amorphous beeswax blocks in the sample. The decrease in T<sub>g</sub> may also be due to soft amorphous beeswax blocks in the molecular chain of the sample.

The drop in storage modulus in the glass transition region is due to the presence of crystalline domains in beeswax. These domains subtly appear to be cross-linked, thus

reducing the storage modulus downfall [71]. This is followed by a sudden descent characterized by a specific modulus value for each sample, thus marking the phenomenon of crystalline phase melting [72]. Up to approximately 90°C the storage modulus obtains a different behavior for each weight percentage of beeswax. However, after this temperature, the behavior of the curves follow a pattern being characterized only the properties of the PDMS, since the beeswax is in a liquid state, having no more interference in the behavior of the curves.

As can be seen in figure 5.2b of the loss modulus, the effect of beeswax on weight is visualized, with the BW8% sample having a loss modulus of 0.23MPa and it was higher for the sample containing the highest percentage of beeswax. Note that there was a greater decrease in the region of 75 to 80°C due to the melting of the crystalline phase of beeswax. Another striking detail regarding beeswax is that the BW8% sample obtained the lowest loss modulus at the end of the 0.018MPa test. Realizing the effect of beeswax in samples with higher loss modules for the highest percentage of beeswax.

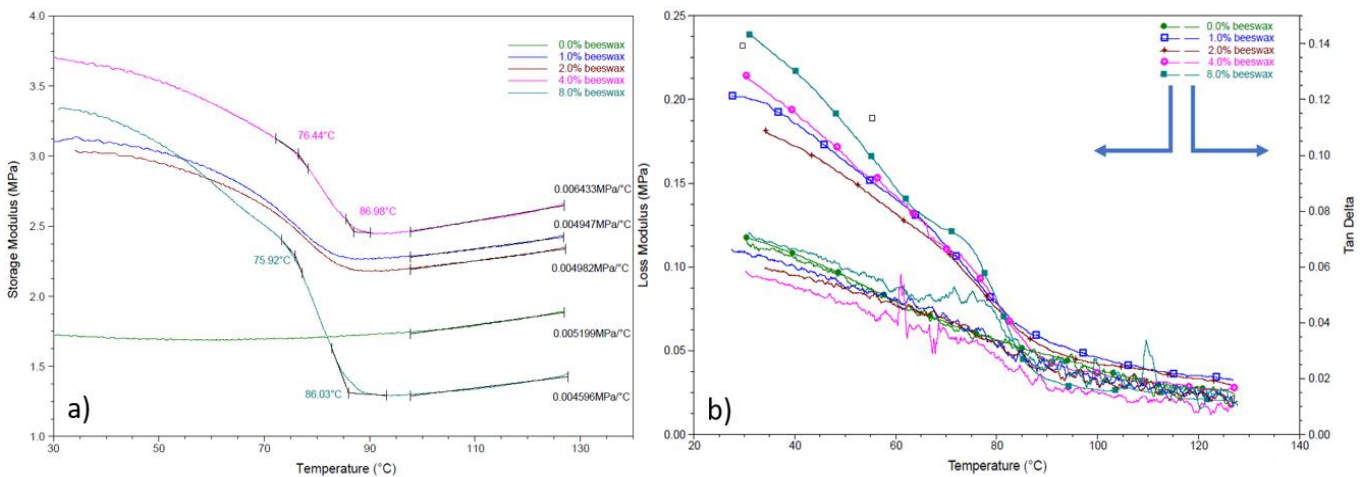


Figure 5.2- Dynamic mechanical analysis. (a) Storage modulus and (b) loss modulus and tan delta.

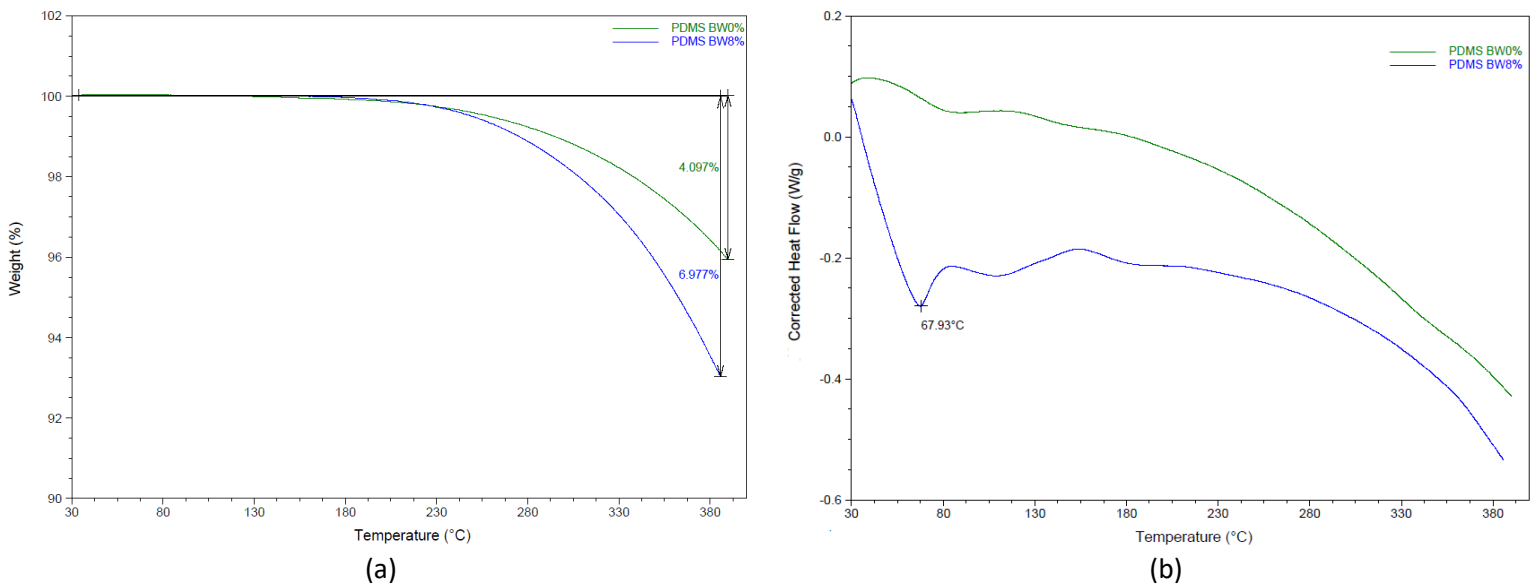
Table 5.3. DMA parameters of composites.

Composição	Storage modulus (MPa) <sub>máx</sub>	Loss modulus (MPa) <sub>máx</sub>	Tan delta
0.0% beeswax	1.72	0.11	-
1.0% beeswax	3.12	0.20	-
2.0% beeswax	3.04	0.18	-
4.0% beeswax	3.70	0.21	0.068
8.0% beeswax	3.34	0.23	0.055

The loss factor showed one peak for the BW4% sample and two peaks for the BW8% sample, one of them being precisely in the glass transition region of the composite at approximately 75 to 80°C.

### 5.3.2 Thermogravimetric test

Gravimetric thermal analysis is an essential parameter to analyze and identify the conduct of thermal stability. The samples of pure PDMS and PDMS with 8% of the beeswax were performed in an inert environment using argon, the result is shown as in figure 5.3. In figure 5.3a it is possible to observe that there is no weight loss until, approximately, 200°C, for both samples. This effect confirms the thermal stability of the composite BW8% and BW0% up to this temperature. The result is consistent with the studies reported in the literature [4], [12]. After 200°C there is a reduction in the weight of the two materials, however, at 390°C it is observed a weight reduction is equivalent to 4% and 7% for BW0% and BW8%, respectively. This 40% greater reduction in the BW8% sample was expected because the BW8% sample contains beeswax in its composition. Due to this considerable weight loss, it is a strong point for thermal stability analysis [73]. A study in the literature shows that the weight loss of beeswax is 100% when subjected to 443°C, it may be for this reason that at approximately 390°C sample BW8% is close to 8% loss [4].



**Figure 5.3- Thermal gravimetric analysis of the pure PDMS and PDMS/beeswax contend 8% of the beeswax.**

In figure 5.3b it is denoted that the peak melting temperatures are similar due to the difference in an inert material. The peak temperature of BW8% in argon is observed at 67.93°C, this value can change depending on the origin and region in which it had access to beeswax, and this value is in accordance with studies reported in the literature [30], [74], [75].

### 5.3.3 Wettability test

The water contact angle (WCA) was determined to assess the hydrophobic nature of the samples, as well as the wettability of their surface. The results for the different measurements of each percentage of beeswax are shown in table 5.4. The measurements of all the percentages made it possible to verify the effect of beeswax on the surface roughness. This effect must be considered the roughness of the mold in which the composite was manufactured.

**Table 5.4- Contact angle values obtained for pure PDMS and composite PDMS/beeswax**

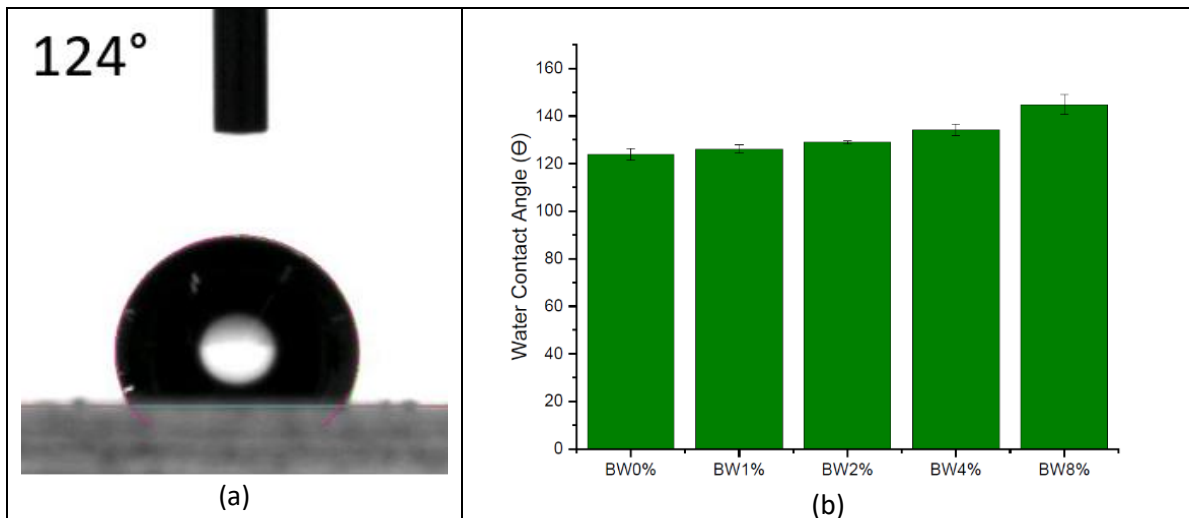
<b>Measurement</b>	<b>BW0%</b>	<b>BW1%</b>	<b>BW2%</b>	<b>BW4%</b>	<b>BW8%</b>
1 [°]	124	128.9	129.3	133.2	145
2 [°]	123.5	126.2	129.1	135.1	144.7
3 [°]	124	126	128.7	136.4	142.3
4 [°]	119	124.7	127.6	130.3	152.2
Arithmetic mean	123.75	126.1	128.9	134.15	144.85
Standard Deviation	2.43	1.76	0.76	2.52	4.27

The analyzed samples represent a hydrophobic behavior for all composites compositions. Figure 5.4a shows pure PDMS sample resulted in a water contact angle equivalent to 124°, which is in agreement with the approximate values reported in the literature [61], [76], [77]. Furthermore, the hydrophobicity of PDMS is related to the methyl/siloxane groups that are compounds present in the chemical structure of the material [9], [78]. A sample with a surface that has a water contact angle less than 90° is considered a hydrophilic surface, when this water contact angle is less than 150° it is considered a hydrophobic surface and when it has a WCA greater than or equal to 150° corresponds to a super-hydrophobic surface [79].

In addition, the addition of beeswax to the PDMS would make the surface super-hydrophobic, presenting an angle of contact with water greater than 150°, due to the formation of domains rich in beeswax inside the sample, which could increase the roughness of the sample. surface and hydrophobicity, caused by the lotus leaf effect [80], [81]. However, as it is possible to analyze the graph in Figure 5.4b, the samples did not exceed 150°, these changes may be due to the reduction of surface roughness and energy, being the main parameters for reducing the wettability of a material [82].

The performance of beeswax in the composites followed the proportion of the amount of beeswax added to the samples, being directly proportional. As the percentage of beeswax increases, the angle of contact with water also increased. However, BW8%

sample has the largest contact angle with water resulting in  $144.85^\circ$ . Compared to BW1% increased the contact angle with water by 15% and 17% compared to pure PDMS.



**Figure 5.4- Results obtained from the wettability test value chart: (a) measurement and (b) value chart.**

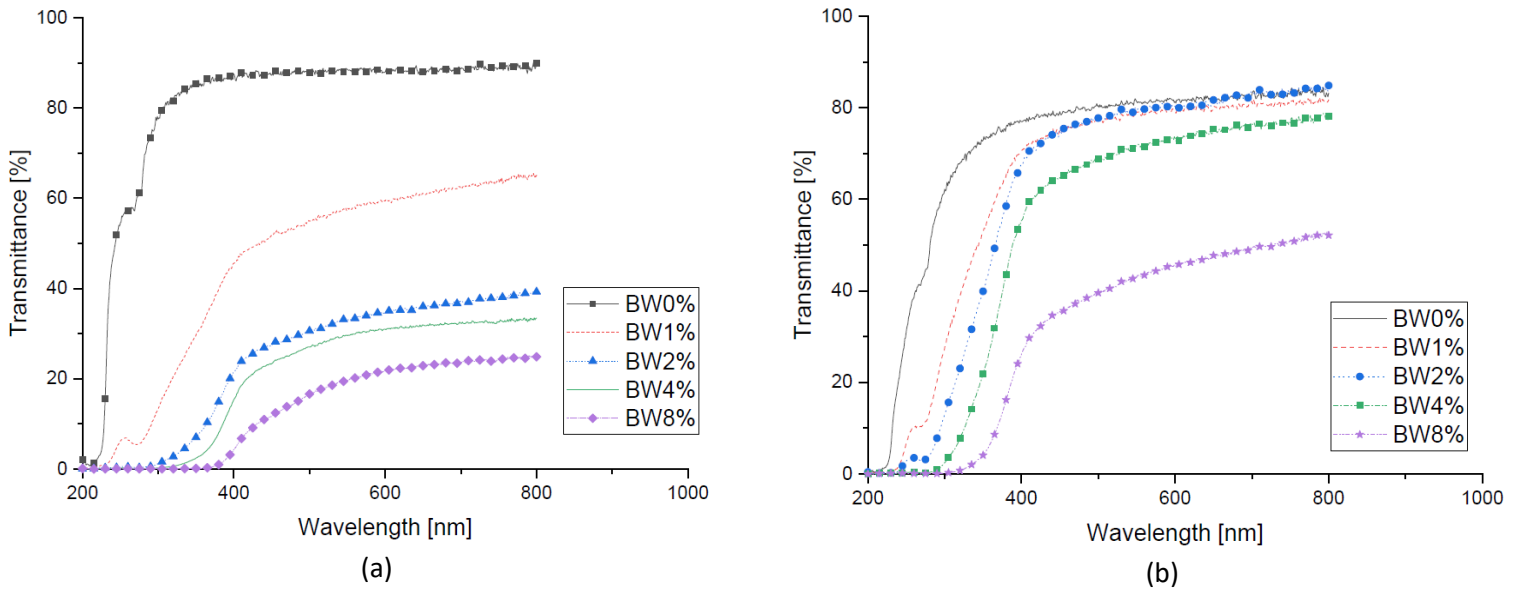
### 5.3.4 Spectrophotometric test

PDMS is a material with high transparency that allows excellent light propagation. To improve its mechanical properties, additives are added in small amounts because the waxes have an opaque effect at room temperature [83]. Pure PDMS showed 89% transparency in the visible region, which is in agreement with studies reported in the literature [41], [84]. However, PDMS mixed with waxes, for example, paraffin wax and beeswax, make it opaque at room temperature. This effect changes course when heat is added to the material, as the temperature increases the material becomes transparent [76], [85]. Other studies with a mixture of PDMS and waxes are also stimulated to transparency by actuation tension, taking a very short time from opaque to translucent [86], [87].

Figure 5.5a shows the spectrophotometry for all samples at room temperature of  $25^\circ\text{C}$ , as mentioned before, the BW0% sample obtained 89% transmittance in the visible region, between 380 and 740 nm. For the BW1% sample, the transmittance value decreased to 64% in the visible region, which is expected due to beeswax having an opaque regiment at room temperature, being compared and consistent with studies reported in the literature [76]. In fact, as the proportion of beeswax increases the transparency decreases, they are inversely proportional. For samples containing a higher proportion of beeswax it is possible to observe that there was a rapid increase in transmittance near the beginning of the visible region, but the transmittance only reached 38%, 32% and 24% for BW2%, BW4% and BW8%, respectively. What was not expected



for the BW2% sample to decrease transmittance by almost 69% for the same range of the visible region, however due to this effect of the wax at room temperature the tendency of the transmissibility is to decrease as the amount of beeswax is added.



**Figure 5.5- Wavelength versus transmittance for different percentages of beeswax at 25°C (a) and 80°C (b).**

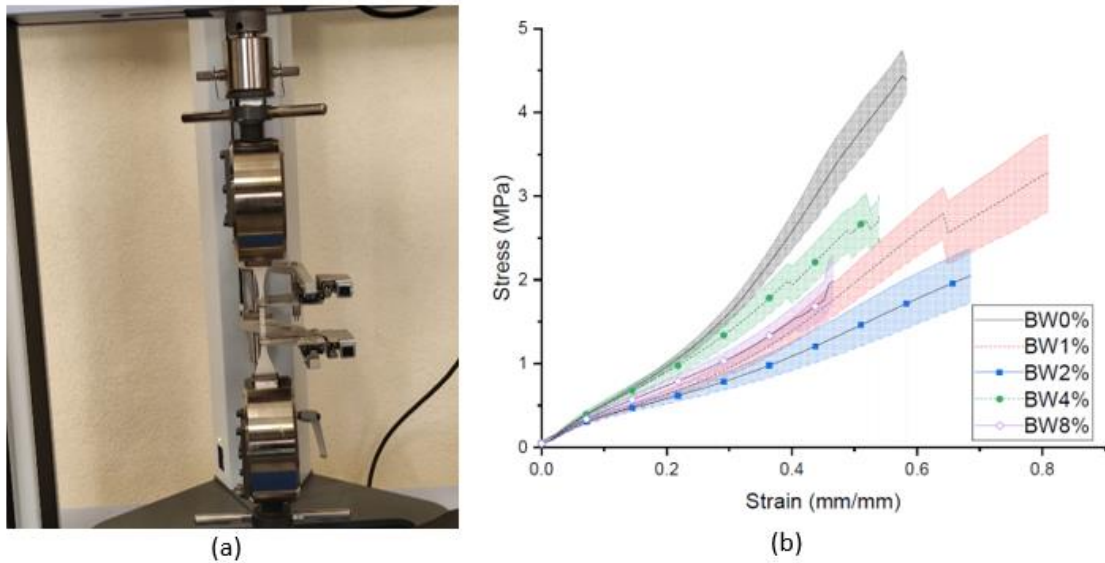
However, when the composites were evaluated at 80°C there was a drastic increase in the transparency of the samples containing beeswax, as shown in Figure 5.5b. The BW1%, BW2% and BW4% samples reached values as high as the pure PDMS in the range of 80% transmittance in the visible region, as shown in table 5.5. BW1% sample obtained a 21% increase when compared to the same sample at 25°C and BW2% sample was surprised with a 54% increase in transmittance when compared to the same sample at 25°C. The BW4% and BW8% samples obtained an increase in the 50% range when compared to 25°C. This effect occurs because waxes in the solid-state provide no scattering of light, which results in opaque, and when the temperature reaches the melting point of the wax, above 65°C, the wax is in a liquid and melted state causing swelling in the PDMS matrix leaving translucent [88]. So far there are no reports of spectrophotometry studies for PDMS samples with higher proportions of beeswax for this test.

**Table 5.5- Comparative results of transmittance carried out to 25°C and 80°C.**

Temperature	BW0%	BW1%	BW2%	BW4%	BW8%
25°C	88.9	63.6	37.8	32.4	23.9
Visible region					
80°C	82.6	81.1	82.9	76.8	50.2
Visible region					

### 5.3.5 Tensile test

Commonly, the mechanical properties of composites are determined by measuring some parameters such as tensile strength and deformation, which are used to measure strength and flexibility [89]. Test parameters were performed in accordance with ASTM D412 as shown in figure 5.6a. The graph of engineering stress versus engineering strain is shown in Figure 5.6b and the data obtained are listed in Table 5.6. The graph shows the arithmetic mean of the tension of each percentage of beeswax together with the pure PDMS and the corresponding standard deviation.



**Figure 5.6- Engineering stress versus engineering strains for composites. (a) Specimen in testing and (b) Stress versus strain graph.**

The standard deviation related to the maximum tensile strength for each sample is approximately 10% of the value obtained by pure PDMS, which is in agreement with the standard deviation reported in the literature [90]. For samples with high standard deviation, it may be due to the equipment's displacement speed, it was necessary to use sandpapers at the ends of the sample, in order to help the samples not slip during the test.

Observing the figure 5.6b, it is possible to visualize a typical behavior of elastic materials with high strain values approximately in the linear region of the samples, which are up to 50% [90]. The BW0% curve refers to the pure PDMS that obtains the highest tensile strength value equivalent to 4.4 MPa in maximum strength, it is slightly below the studies reported in the literature [76]. Sample BW1% containing 1% by weight of beeswax showed a 35% reduction in maximum tensile strength compared to BW0% and an increase in the maximum strain of approximately 37%, in response it may be due to

the ductile behavior of the beeswax after an applied stress [30], [52], so this effect can justify the reduction in maximum tensile strength and increase in maximum strain [22]. Another attempt to explain the decrease in tensile strength is that the addition of beeswax to the PDMS affects the cohesive forces of the net, resulting in a more elastic composite [58]. When using synthesized filtration membranes, the increase in the PVCA/PDMS mixture also increases the tensile strength of the blend [36]. Other studies indicate that PDMS when crosslinked with the curing agent form trapped tangles to improve the network, thus increasing the mechanical properties, consistent with the present study [91], [92]. The composite BW1% manufactured in this work had higher maximum strength than compared to our other study, carried out previously, having the same proportion in weight of beeswax, this value was 12% higher [76].

The BW2% curve has a similar behavior with the BW1% curve but obtained a reduction in the maximum tensile strength of 59% compared to BW1% and 116% compared to pure PDMS. This effect may have occurred due to the concentration of beeswax particles in the macromolecular region. For the samples BW4% and BW8% obtained an unexpected behavior, drastically reduced deformation and an increase in tensile strength, not expected. The deformation values were reduced by more than 27% for BW4% and 46% for BW8% compared to the BW2% sample, which was expected to behave in this condition. This behavior of the BW4% and BW8% samples may be due to the concentration of beeswax particles in the intercellular and macromolecular region, as described above, but this effect should act in the opposite way since beeswax promotes increased tenacity [52].

An experimental study found that fatty acids confer ductile properties to beeswax because experimental removal of fatty acids from beeswax causes a lowering of the elastic limit [93]. However, the addition of beeswax improves the tenacity of the composite [52]. Johnston et al observed a relationship between the modulus of young and the curing temperature of the samples, revealing that the curing temperature is directly proportional to the modulus of young, so to increase the elastic modulus values just increase the curing temperature [90].

This behavior for pure PDMS stress/strain curves have been reported in the literature measuring approximately 5MPa[90]. The behavior for stress/strain curves of PDMS with the addition of 1% beeswax measuring approximately 2.5MPa has been reported [76]. Pure beeswax has been reported to have a maximum tensile strength of

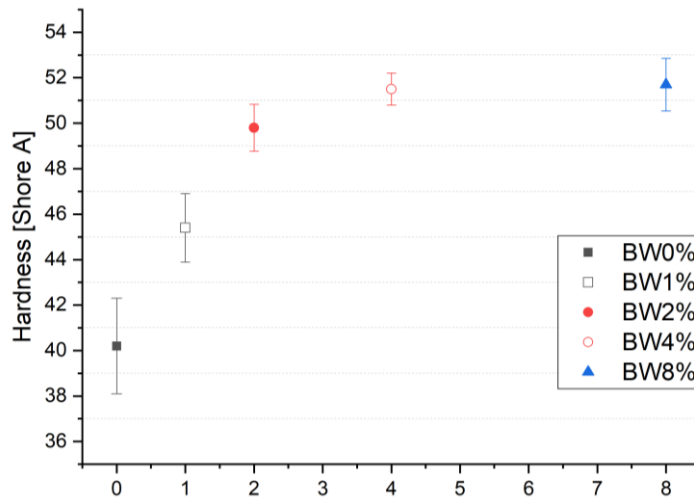
0.75MPa [30]. Until the present study, the measurement of tensile strength with higher proportions of beeswax was not reported.

**Table 5.6- Mechanical properties of PDMS from stress/strain testing.**

<b>Composites</b>	<b><math>\sigma_{M\acute{a}x}</math> (MPa)</b>	<b><math>\epsilon_{M\acute{a}x}</math> (mm/mm)</b>
BW0%	4.43	0.58
BW1%	3.27	0.81
BW2%	2.05	0.68
BW4%	2.74	0.53
BW8%	1.99	0.46

### 5.3.6 Hardness test

Figure 5.7 shows a summary of all results obtained in the hardness tests for pure (BW0%) PDMS and its composites with 1, 2, 4, and 8% of beeswax addition. The points were positioned on the horizontal axis (abscissa) according to the amount of wax used. In addition, the standard deviation values were also plotted.



**Figure 5.7- Results: Hardness Shore A tests.**

Although the hardness always increases directly proportionally to the increase in the wax amount, the results can be analyzed in two groups, the first one regarding small additions (between 0 and 2%) and the second for materials with the highest amount of wax (4 and 8%).

In the first group, pure PDMS presented results very close to those shown in the manufacturer's datasheet, which is 44 Shore A [67]. Furthermore, as the amount of wax is increased, the increase in hardness presents a linear relationship (coefficient of determination  $R^2=0.9877$ ) until reaching 4% of beeswax.

**Table 5.7- Results of hardness test.**

<b>Measurement</b>	<b>BW0%</b>	<b>BW1%</b>	<b>BW2%</b>	<b>BW4%</b>	<b>BW8%</b>
Average	40.2	45.4	49.8	51.5	51.7
Standard deviation	2.098	1.506	1.033	0.707	1.160

Above that value, at the second sample group, the average values were very close, which means that considering the standard deviation presented, by doubling the amount of wax, from 4 to 8%, the hardness was not affected.

## **5.4 Conclusions**

The present study was aimed at investigating the effect that beeswax has on PDMS, mainly hydrophobicity. This composite with various proportions of beeswax is little explored in the literature, in addition, tensile, hardness, spectrophotometric, DMA, TGA and wettability tests were performed, which makes these results obtained are seriously promising.

The mechanical tensile properties of the BW1% sample suffered a 35% decrease in tensile strength and a 37% improvement in deformation, as expected due to the ductile properties of beeswax and when compared to pure PDMS (BW0%). The shore A hardness test revealed a 27% increase in the BW8% sample compared to BW0%. These data show that the composite has increased tenacity with the possibility of applications in food protection films.

The transmittance properties of the BW1% sample showed a 28% decrease compared to pure PDMS at room temperature in the visible region (wavelengths around 740nm). However, when these samples were subjected to the temperature rise to approximately 80°C there was a 30% improvement in the transmittance of the BW1% sample compared to ambient temperature, making it as transparent as pure PDMS in the visible region. Due to these results, there is a wide range of applications in films with the ability to change the translucency.

The thermogravimetric analysis tests showed that even when high amounts of up to 8% of beeswax, the thermal behavior is stable up to approximately 200°C, making a material thermally stable and with potential application in environments that require high temperatures. The Dynamic mechanical analysis test revealed that the storage modulus is directly proportional to the amount of beeswax in the composition up to a value of 8% by

weight. Sample BW8% showed an improvement of almost 100% when compared to BW0%, making the composite more elastic for coating film applications.

The hydrophobic properties of PDMS were improved with the addition of beeswax, this result of 124° for pure PDMS reached 145° for PDMS with 8% by weight of beeswax (BW8%). This property obtained an improvement of 17%, thus being a hydrophobic surface, however some measurements surpassed 150° which indicates a superhydrophobic surface. These results have enormous potential for applications that require anti-fog, water repellent and self-cleaning properties. Concluding that it is possible to increase the hydrophobic and superhydrophobic properties of PDMS with the addition of beeswax.

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# Chapter 6

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## Conclusion and Future Directions

### 6.1 Conclusions

In this work the objectives were to develop equipment to facilitate and make the manufacturing process of PDMS with waxes more efficient, it was also the characterization of the beeswax used to manufacture the samples and finally, the samples were manufactured and the effect of the addition of beeswax on the properties of polydimethylsiloxane (PDMS).

The development of equipment to optimize the composite manufacturing process gave rise to a multifunctional vacuum chamber that is capable of carrying out all stages of the manufacturing process individually or simultaneously. The use of the MVC was satisfactory on the requirement of ease of operation with full control of the usage parameters, namely, temperature control, mixing speed control, and internal pressure. The results obtained from the use of this equipment was an improvement in the dispersion of beeswax in the PDMS matrix and there was a relevant improvement in the processing time equivalent to 150% less when compared to the conventional method.

Beeswax is a material synthesized from honey sugars and secreted in glands in a liquid form located in the abdomen of the bee. However, this process undergoes changes in its composition due to various climatic and macro-regional factors and the location of fixation of the hives. Therefore, the characterization of beeswax is of High factor for the affected results in the properties. The Fourier-transform infrared spectroscopy test was performed comparing a purified beeswax, the result revealed that the beeswax used in this work has 99.6% purification, and the rest was not compatible with the adulterants, reinforcing that the beeswax there was no tampering.

The manufactured PDMS/beeswax composite was carried out for mechanical, optical and wettability characterization. In tensile testing, the composite BW1% suffered a 35% decrease in tensile strength and a 37% improvement in deformation when compared to BW0%. On shore A hardness test revealed an increase of 27% for the BW8% sample. These data show greater tenacity with the possibility of applications in food packaging.

Optical properties showed a 30% increase in transmittance in the visible region when compared to room temperature and 80°C for BW1%, being as transparent as pure PDMS. In thermogravimetric analysis, the composite showed thermal stability up to 200°C. After these data, the composite has a wide range of applications in transparent films with the ability to change the translucency with a temperature trigger and also in applications in coating films for photovoltaic panels.

The dynamic mechanical analysis test showed that the storage modulus is directly proportional to the amount of beeswax in the composite, and the BW8% sample increased the storage modulus by 100% when compared to the BW0%. Thus, making the composite more elastic.

The hydrophobic properties of PDMS were improved with the addition of beeswax, while the BW0% had 124° of contact angle with water, the sample BW8% had a contact angle with water equivalent to 145°, with an improvement of 17%, however, some measurements of the water contact angle revealed greater than 150° making the surface superhydrophobic. This data has enormous potential in surface applications that require anti-fog, water repellent and self-cleaning properties.

## **6.2 Future Directions**

The present study obtained the ability to analyze many tests and proportions of beeswax by weight in the PDMS matrix, however, it is proposed to analyze the interface between beeswax and PDMS, as well as the dispersion of beeswax in PDMS with the aid of a microscope.

A very relevant factor in question would be to increase the amount of beeswax impregnated in the PDMS matrix and carry out the tests for a complete analysis of the composite with a high content of beeswax. Furthermore, development for possible applications can be carried out from this manufactured composite.

Develop new composites based on PDMS and other elements in order to carry out the same number of tests in the present dissertation.

Conduct a study with different beeswax from different regions and climatic conditions.

# Appendix A

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## List of Publications

The development of this dissertation resulted in: publications in international journals and publications in indexed journals.

### Publications

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Ariati, R. et al., 2021. PDMS characterization and its applications: A review. Article in submission process in Journal Polymers, MDPI.

Ariati, R. et al., 2021. Multifunctional low-cost vacuum chamber to manufacturing PDMS based composites. Article in submission process in Journal Machines, MDPI.

Ariati, R. et al., 2021. Characterization properties of Beeswax. Article in submission process in Journal of Composites Science, MDPI.

Ariati, R. et al., 2021. Study mechanical, optical properties and wettability of PDMS/Beeswax composite. Article in submission process in Journal of Materials Science, Springer.