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Rami Alawar, Pierre-Jacques Liotier, Romain Ravel, Monica Francesca Pucci. Estimation of the surface tension and dispersive and polar components of polymers as a function of temperature for composite manufacturing applications. *Polymer Testing*, 2026, 156, pp.109112. <10.1016/j.polymertesting.2026.109112>. <hal-05517297>

HAL Id: hal-05517297

<https://imt-mines-ales.hal.science/hal-05517297v1>

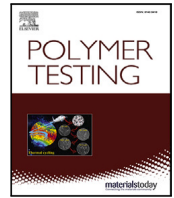
Submitted on 18 Feb 2026

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Estimation of the surface tension and dispersive and polar components of polymers as a function of temperature for composite manufacturing applications

Rami Alawar^{a,*}, Pierre-Jacques Liotier^b, Romain Ravel^b, Monica Francesca Pucci^{a,*}

^a *Polymers Composites and Hybrids (PCH), IMT Mines Alès, Alès, France*

^b *LMGC, IMT Mines Alès, Univ Montpellier, CNRS, Alès, France*

ARTICLE INFO

Keywords:

Surface tension
Polarity
Dispersive component
Temperature
Molten thermoplastic
Surface characterization

ABSTRACT

Understanding adhesion between fiber and matrix at elevated temperatures is essential for improving the mechanical performance of polymer-based composites, especially with thermoplastic matrices. However, detailed characterization of polymer surface tension and its polar and dispersive components as a function of temperature remains limited. In this work, reliable methods were set using the Wilhelmy plate and pendant drop approaches to investigate these properties against temperature. First, experimental procedures were developed, optimized, and validated through cross-comparison with reference liquids of known surface tension and components. Accurate and reproducible measurements were secondly achieved across a range of elevated temperatures for liquid polymers (polyethylene glycol, bio-based epoxy) and for molten thermoplastics (polypropylene, polylactic acid). The results reveal a linear decrease in surface tension with increasing temperature and contribute to a better understanding of fiber wetting phenomena. Additionally, a procedure was set to determine polymer dispersive and polar components as a function of temperature. Due to the volatility and thermal limitation of n-hexane used in interfacial tension measurements, alternative probe liquids were systematically evaluated. Silicone and paraffin oil were identified and validated as suitable replacements, enabling reliable measurements with polymers at high temperatures. These key findings demonstrate robust methodology for high-temperature surface characterization and provide essential data to understand fiber–matrix adhesion under realistic processing conditions.

1. Introduction

Ongoing research in the field of composite materials is placing great emphasis on reducing their environmental footprint by reevaluating their entire life cycle. Environmental evaluations typically emphasize three key aspects: the life cycle of each constituent material, the energy efficiency involved in the manufacturing process, and the end-of-life management of the composites. Increasing awareness of the environmental consequences associated with non-recyclable and non-biodegradable plastics, particularly at the end-of-life stage, has generated strong interest in developing nature-based and recyclable alternatives to synthetic materials. However, the development of such sustainable composites requires a careful balance between enchaining their recyclability and biodegradability while maintaining the essential mechanical characteristics necessary for practical applications [1,2]. Scientific research indicates that natural reinforcing fibers, derived from animal and plant sources, present promising alternatives to traditional reinforcing fibers such as glass fibers and carbon fibers. At the

matrix level, thermosets can be replaced by thermoplastics, as the latter can be repeatedly processed by heating to the processing temperature and recycled directly into new structural shapes [3–5].

Nowadays, thermoplastic composites are increasingly being used in aerospace, automotive, and marine industries, thanks to their enhanced heat resistance, durability, toughness, strength, and sustainability in high-temperature environments. For such large-scale industrial applications, composite parts are commonly manufactured using automated tape laying techniques using thermoplastic tapes [6–8]. Changing the nature of fibers and matrices requires total control of composite manufacturing and above all the fiber–matrix interface. In composite manufacturing, both the viscosity and surface tension of a polymer significantly impact the capillary forces which plays a dominant role in dynamic wetting during composite processing [2,9,10]. As temperature is a primary factor affecting the magnitude of surface tension, evaluating this parameter is essential, especially when dealing with thermoplastics that are processed at elevated temperatures [11].

* Corresponding authors.

E-mail addresses: rami.alawar@mines-ales.fr (R. Alawar), monica.pucci@mines-ales.fr (M.F. Pucci).

<https://doi.org/10.1016/j.polymeresting.2026.109112>

Received 27 November 2025; Received in revised form 6 January 2026; Accepted 8 February 2026

Available online 9 February 2026

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Another critical parameter in composite manufacturing is the adhesion between the fiber and the matrix [12,13]. Adhesion between the two phases can be predicted by comparing the ratio of their dispersive to polar components. A greater similarity in these ratios typically indicates stronger interfacial interactions. In other words, the adhesion between the fiber and the matrix improves when their polar and dispersive components are closely matched [14–16]. Therefore, evaluating the surface energy components of polymers as a function of temperature is essential for a deeper understanding of fiber/matrix adhesion.

Significant efforts have been dedicated to measuring the surface tension of liquids and its individual components, leading to the development of various experimental techniques. Among the most widely used methods reported in the literature are the Wilhelmy plate and the pendant drop methods. The Wilhelmy plate method is a force-based technique that measures surface and interfacial tension by evaluating the force exerted on a thin plate partially immersed in the liquid. In contrast, the pendant drop method employs an optical approach, analyzing the shape of a suspended droplet and applying the Young–Laplace equation to determine surface and interfacial tension [11,17–20].

The originality of this work lies in the deep investigation of surface tension and the polar and dispersive components of polymers at elevated temperatures using both the Wilhelmy plate and pendant drop methods. Reliable and optimized procedures were developed for each technique, enabling accurate measurements at elevated temperatures for liquid polymers (polyethylene glycol, bio-based epoxy) and for molten thermoplastics (polypropylene, polylactic acid). Notably, despite the fundamental importance of the surface energies in polymer processing, up to our knowledge there is no reported data in literature regarding the temperature dependence of the polar and dispersive components of polymers. Thus, this study has been designed to address this critical gap. Knowing the thermal limitations of n-hexane in interfacial tension experiments, that are essential for surface energy components determination of a polymer, alternative probe liquids were systematically evaluated and validated to ensure compatibility with high-temperature conditions. This comprehensive approach offers new insights and practical methodologies for characterizing polymer interfaces under realistic processing environments, advancing the current understanding of polymer surface science, specifically by demonstrating the influence of surface tension variations on dynamic wetting and the role of surface energy component variation in controlling the adhesion between the fibers and matrices.

2. Materials and methods

2.1. Materials

2.1.1. Test liquids and polymers

The selected test liquids were water, n-hexane and ethylene glycol, which were provided by Merck. Table 1 presents the surface tension γ_L , the polar γ_L^p and dispersive γ_L^d components, as well as the density ρ of the liquids tested at room temperature. These liquids were used as reference liquids to determine their surface tension using two different methods presented in Section 2.2 allowing to validate the methods and assess the repeatability and reliability of results. Moreover, since n-hexane is a totally dispersive-low density liquid, it was also used to determine the polar and dispersive components of the other testing liquids and polymers.

As for the liquid polymers, polyethylene glycol 300 (CARBOWAX PEG 300) with a density of 1.125 g/cm³ at 20 °C, silicone oil (XIAMETER PMX-200 100 cSt) with a density of 0.964 g/cm³ at 25 °C that are provided by DOW, paraffin oil bought from ThermoFisher Scientific with a density of 0.88 g/cm³ at 20 °C, and the bio-based epoxy resin (SR InfuGreen810) purchased from Sicomin with a density of 1.16 g/cm³ at 20 °C were first used to study their surface tension at both room and

Table 1

Test liquids characteristics at 20 °C [20].

	ρ (g/cm ³)	γ_L^p (mN/m)	γ_L^d (mN/m)	γ_L (mN/m)
n-Hexane	0.659	0.0	18.4	18.4
Water	0.998	51.0	21.8	72.8
Ethylene glycol	1.113	19.0	29.0	48.0

elevated temperatures, as well as their polar and dispersive components at room temperature. Furthermore, since silicone oil and paraffin oil are totally dispersive low-density liquids, they were also used to study the polar and dispersive components of the polymers.

Regarding the thermoplastic polymers, polypropylene (PP 48M10) supplied by Sabic with a density of 0.905 g/cm³, and polylactic acid (PLA - Ingeo Biopolymer 3251D) purchased from NatureWorks with a density of 1.24 g/cm³ were selected. Polypropylene was chosen because of its ability to be recycled [21], while polylactic acid was selected due to its biopolymer nature [22].

2.2. Methods

2.2.1. Polymer's polar and dispersive component determination

To determine the polar and dispersive components of the surface tension of polymers, the well-known Owens and Wendt equation (Eq. (1)) was employed [20].

$$\gamma_{L1L2} = \gamma_{L1} + \gamma_{L2} - 2(\gamma_{L1}^d \gamma_{L2}^d)^{0.5} - 2(\gamma_{L1}^p \gamma_{L2}^p)^{0.5} \quad (1)$$

This equation relies on two liquids. The first liquid (L1) is required to be purely dispersive, meaning its total surface tension is equal to the dispersive component ($\gamma_{L1} = \gamma_{L1}^d$). Additionally, L1 should have a density lower than that of the tested liquid. The second liquid (L2) is the polymer under investigation, with a surface tension denoted by γ_{L2} . The interfacial tension between the two liquids is represented by γ_{L1L2} . In this context, the superscripts *p* and *d* refer to the polar and dispersive components of surface tension, respectively.

Knowing that the first liquid should be purely dispersive ($\gamma_{L1}^p = 0$), the Owens and Wendt equation (Eq. (1)) can be rearranged to yield the following form:

$$\gamma_{L2}^d = \frac{(\gamma_{L1} + \gamma_{L2} - \gamma_{L1L2})^2}{4\gamma_{L1}^d} \quad (2)$$

Using Eq. (2), the dispersive component of the tested polymer can be determined. Subsequently, the polar component can be determined using the Owens–Wendt theory, which decomposes the surface tension into two contributions: a polar component and a dispersive component, as shown in Eq. (3) [14].

$$\gamma_{L2} = \gamma_{L2}^p + \gamma_{L2}^d \quad (3)$$

Thus, the Owens and Wendt method can be employed to determine the polar and dispersive components of the tested polymers. However, it requires prior knowledge of the surface tension of the two liquids (γ_{L1} and γ_{L2}) as well as the interfacial tension between them (γ_{L1L2}).

2.2.2. Tensiometer - Wilhelmy Plate (WP) method

One Step Wilhelmy Plate Procedure

The Wilhelmy plate method was applied using a Krüss K100 SF Tensiometer apparatus in order to determine the surface tension of the utilized liquids. The plate was first immersed in the liquid contained in a thermally controlled bath with an immersion depth of 3 mm. The stage is then moved down to allow the micro balance to measure the meniscus weight *m* formed by the liquid around the plate [20,23,24]. Knowing that the plate is made of platinum, thus allowing a zero contact angle with the liquids, and knowing the wetting parameter *p* of the plate [25], the surface tension of the liquid can be derived using the Wilhelmy relationship:

$$F_c = ma = p\gamma_L \cos \theta \quad (4)$$

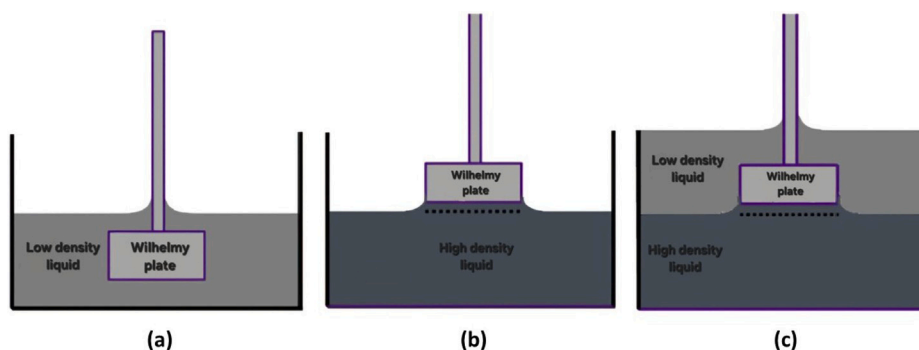


Fig. 1. Interfacial tension test steps by WP method.
Source: Taken from [20].

where F_c is the capillary force, a is the acceleration due to gravity, θ is the contact angle and γ_L is the surface tension of the liquid.

The thermally controlled bath is heated using a JULABO F12-MA heating circulator. This heating circulator allows the heating of the liquid up to 200 °C. However, considering the heat losses through the tubing connection between the tensiometer and heating circulator, the maximum temperature of the tested liquid achieved was measured to be 120 °C.

Three steps Wilhelmy Plate procedure

A three step procedure was applied on the same Krüss K100 SF Tensiometer apparatus to measure the interfacial tension between the tested liquids and a reference low-density liquid.

First, the Archimedes' force is measured on the Wilhelmy's plate that is totally immersed in the low density liquid (Fig. 1a). In the second step, the surface tension of the tested liquid is measured (γ_{L2}), using the simple principle mentioned previously (Fig. 1b). Finally, the low-density liquid is added on the top of the tested liquid (Fig. 1c). The meniscus weight m formed at the interface is measured by the micro balance, and the interfacial tension between the two liquids (γ_{L1L2}) is then obtained [20].

2.2.3. Goniometer - Pendant Drop (PD) method

The pendant drop method was applied using a Krüss Drop Shape Analyzer (DSA 30) to determine the surface tension of the tested liquids. This is a promising method for precisely calculating the surface and interfacial tension of liquids at both room and elevated temperatures [26, 27]. The setup required for measuring the surface tension consists of a high-resolution camera, a dosing syringe, a light source, a diffuser, and computerized software as shown in the schematic representation of the setup in Fig. 2a.

Some important factors to be considered while conducting the experiments are the drop volume to ensure that the gravitational force is not negligible, and the symmetrical drop shape which can be achieved by using a vertical needle [27]. At equilibrium, a pendant drop follows the Young-Laplace equation (Eq. (5)), which describes the relationship between Laplace pressure across an interface, the interface's curvature, and the interfacial tension:

$$\gamma_L \left(\frac{1}{R_1} + \frac{1}{R_2} \right) = \Delta P \quad (5)$$

where R_1 and R_2 are the principal radii of curvature, γ_L is the liquid surface tension, and ΔP is the pressure difference between that inside the drop and that of the surrounding medium [28]. It should be noted that the Young-Laplace equation for a pendant drop can only be solved numerically. Therefore, in this study, the OpenDrop software is utilized to determine the surface tension of the liquid [27]. The Young-Laplace equation is iteratively solved to achieve the best fit that converges with the drop's outer profile. For the best fit, the value of the dimensionless Bond number B_o and the radius of curvature at the drop apex R_0 are determined. Using Eq. (6), the surface tension γ_L can then be

calculated, where $\Delta\rho$ is the density difference between the drop density ρ_d and the continuous density ρ_c [27].

$$B_o = \frac{\Delta\rho g R_0^2}{\gamma_L} \quad (6)$$

At room temperature, the surface tension of the liquids was measured with air as a surrounding medium. While for surface tension measurement at elevated temperatures, the heating chamber shown in Fig. 2b is used. With this heating chamber the maximum temperature that can be set is 400 °C, but considering the heat losses, the maximum achieved temperature was measured to be around 340 °C. For thermoplastic polymers, the tests were conducted under a flowing atmosphere of nitrogen at a rate of 40 ml/min, while for the liquid polymers the measurements were carried out with air as a surrounding medium (see Fig. 3).

As shown in Eq. (6), the surface tension measurement is dependent on the density of the polymer. Thus, the density of the polymers at each tested temperature is required to post treat the images and to calculate the value of surface tension. For this purpose, the sessile drop (Ellipse fitting) and pendant drop (Young Laplace fitting) methods were employed to measure the drop volume through profile fitting of the drop shape, using the ADVANCE software (version 1.9-03) provided by Krüss. Then, using a Sartorius analytical balance the mass of the corresponding drops were measured and the densities were then calculated as a function of temperature.

As for the interfacial tension tests, a glass vessel provided by Krüss was used. The vessel was filled with the selected low-density liquid, then the needle was immersed in this liquid. Finally, drops of the tested liquid were dosed and captured so that they can be post-treated. Fig. 2c shows an interfacial tension test between ethylene glycol (high-density liquid) and n-hexane (low-density liquid).

2.2.4. Differential Scanning Calorimetry (DSC)

The differential scanning calorimetry (DSC) tests were performed using a PerkinElmer DSC 8000 apparatus. The aim of these tests was to determine the melting temperature of the thermoplastics. A programmed temperature cycle consisting of a controlled heating step to melt the polymer, followed by a controlled cooling step to erase any thermal history within the polymer, then followed by a final controlled heating step was adopted. The melting point of the thermoplastic was recorded from the endothermic peak resulting from the second heating step in the DSC curve. Three tests for each polymer were conducted with a heating and cooling rate of 10 °C/min under a flowing high-purity nitrogen atmosphere (99.999%) with a purge rate of 20 ml/min.

2.2.5. Thermogravimetric analysis (TGA)

The PerkinElmer TGA 8000 apparatus was used to perform the thermogravimetric analysis (TGA). Using this apparatus, the thermal degradation of the tested liquids and thermoplastics polymers were

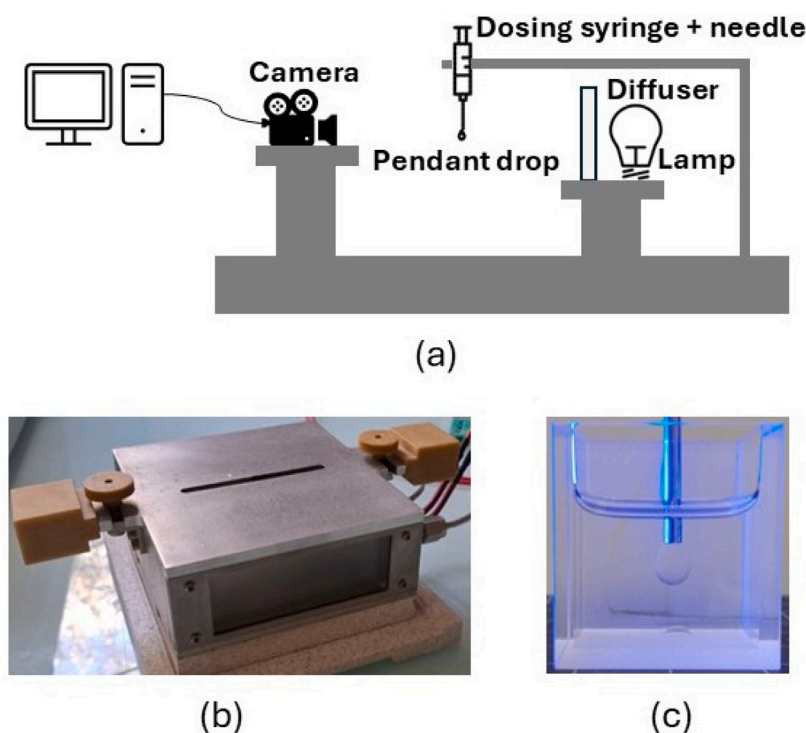


Fig. 2. (a) Schematic representation of goniometer setup, (b) Heating chamber, (c) Glass cuvette for interfacial tension test by PD method.

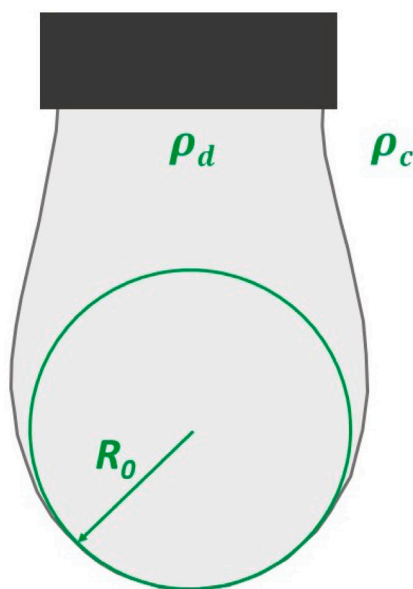


Fig. 3. Pendant drop schematic representation with the essential variables used by the computational software to determine the Bond number.

studied over a wide thermal range. A programmed temperature cycle consisting of a 20 °C temperature scan, followed by an isothermal step of 10 or 15 min was followed, and these two steps were repeated until the entire thermal range was covered. The waiting times were chosen to compensate for the time required to perform surface tension tests (a test duration of 3 min was sufficient to reach a stable meniscus force when using the Wilhelmy plate method for liquid polymers. In contrast, for the pendant drop method, 7–11 min were required to melt the thermoplastics and to form a stable pendant drop). A heating rate of 10 °C/min was used. This temperature profile was chosen to be close

to the time needed to record surface tension measurements at elevated temperatures, especially for thermoplastic polymers, allowing them to melt, form a pendant drop, and achieve equilibrium. The tests were conducted under a flowing atmosphere of nitrogen or oxygen at a purge rate of 20 ml/min. As for the thermal ranges, PEG 300 and the bio-based epoxy resin were studied up to 200 °C, while silicone oil and paraffin oil were analyzed up to 400 °C. The thermoplastic polymers, PP and PLA, were tested up to 500 °C. The liquid polymers were studied under flowing atmosphere of oxygen, while the thermoplastic polymers were examined under the flowing atmosphere of nitrogen. Each polymer was subjected to a single test at each isothermal condition, once with a 10-minute isothermal step and once with the 15-minute isothermal step.

Following the above adopted program, the polymers sample weight percentages versus temperature were monitored. A value of 5% of weight decomposition of the tested polymer was considered as the onset of polymer degradation, as this level of weight loss can lead to a significant reduction in the mechanical properties of the tested material [29,30].

Fig. 4 shows the thermogravimetric behavior of silicone oil under the flow of oxygen. The black plot shows the silicone oil behavior with 10 min isothermal steps, while the red plot shows its behavior with 15 min isothermal steps. Considering the 5% weight loss, thus at 95% of the silicone oil sample weight percentage, the degradation temperature can be determined. As seen in Fig. 4, the silicone oil starts to degrade at a temperature of 300 °C.

3. Results and discussion

3.1. Pendant drop volume and needle size effect on the surface tension measurement

Although the pendant drop is a straight forward technique for determining the surface tension of liquids, it was essential to evaluate the influence of some parameters in order to develop a reliable experimental procedure that ensures high accuracy in results, that can be then extended to thermoplastics.

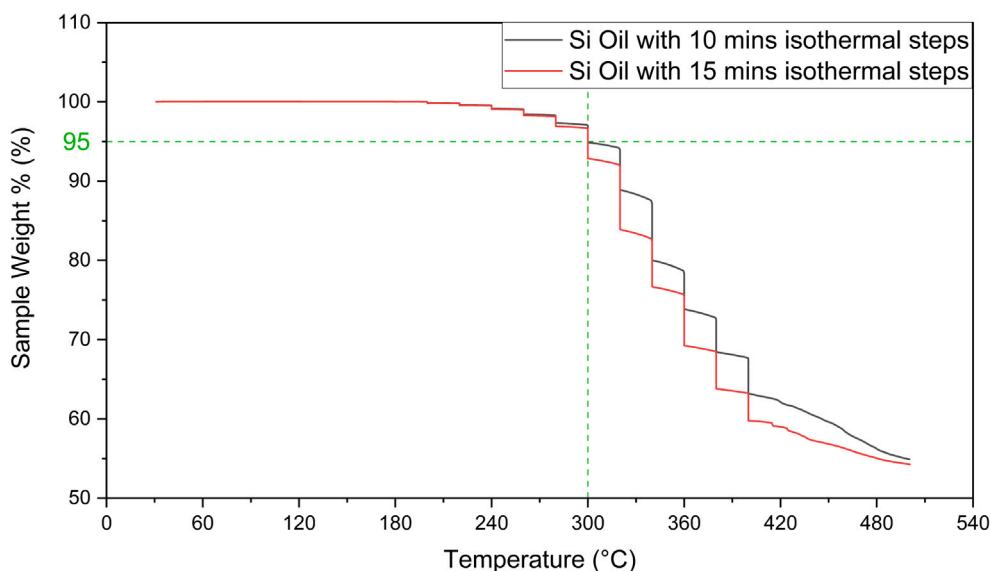


Fig. 4. Silicone oil TGA under oxygen flow.

First, the effect of drop volume on the measured surface tension was examined. Using a 1 mm needle diameter, the surface tension of water was measured at room temperature by varying the volume of the pendant drops formed between 8 μL and 17.5 μL . Fig. 5 presents the average results from three tests conducted at each selected drop volume. It is evident that for volumes below 14 μL , the calculated surface tension values of water deviate significantly from the theoretical value of 72.8 mN/m at 20 $^{\circ}\text{C}$, and the precision of the results is low as indicated by the high standard deviation values. In contrast, for drop volumes greater than 15 μL , the results show higher accuracy, closely aligning with the theoretical value, and exhibit higher precision, as indicated by the low standard deviation indicating strong repeatability.

The variation of surface tension with respect to the drop volume has been reported in the literature by various authors [31–33]. In particular, the results obtained in this study are in good agreement with those reported by J.D. Berry et al. in [27], where it was observed that small drop volumes would result in a very low Bond number B_o during the drop profile fitting. This is due to interfacial forces dominating gravitational forces, leading to a nearly spherical drop shape seen in Fig. 5 at 8 μL . While for larger drop volumes, the effect of gravity, which is an essential factor to calculate the Bond number B_o (Eq. (6)) becomes more significant. This results in improved Bond number estimation, and consequently better surface tension determination.

On the other hand, the effect of needle size on the measured surface tension was investigated. As shown in Fig. 6, the surface tension of water and ethylene glycol were measured using the pendant drop method at room temperature, while varying the needle diameter between 1 mm and 3 mm. The results indicate that the selection of the needle size is adequate in determining the surface tension. For both water and ethylene glycol, the most accurate surface tension values were obtained using a 2 mm needle diameter. After calculating the capillary length l_c of the tested liquids, using Eq. (7), which relates gravity and surface tension, it was observed that needle diameters larger than the capillary length of the tested liquid are not suitable for accurate measurements. This explains why the 3 mm needle diameter was less effective. Therefore, knowing in approximation the value of the capillary length can be useful for selecting the optimal needle size for surface tension measurement using the pendant drop method.

$$l_c = \sqrt{\frac{\gamma_L}{\Delta\rho g}} \quad (7)$$

Additionally, visual observation of the drop shape reveals that very small needle sizes results in nearly spherical drops, even when the drop volume approaches maximum the needle can support. This leads back to the previously discussed issues related to low Bond numbers. Conversely, for very large needle sizes (such as 3 mm diameter), the drop shape may resemble a “U” (as seen with ethylene glycol), making it more difficult to perform accurate profile fittings during image post-processing. Moreover, when attempting to form a larger pendant drop, necking phenomena can occur below the needle (as observed with water using the 3 mm needle). This causes the drop to enter a non-equilibrium state due to its tendency to detach quickly from the needle, compromising the reliability of the measurement. It should be noted that a needle diameter of 2 mm was found to be optimal for the liquids investigated in this study. For other liquids, the optimal diameter may vary depending on factors such as capillary length and drop shape.

3.2. Validation and comparison of surface tension methods

Water, ethylene glycol, and n-hexane were selected as test liquids because they are well-documented in literature, with known surface tensions values and well-established polar and dispersive components. First of all, the surface tension of these three liquids were measured using both the Wilhelmy plate and pendant drop method, with three tests conducted for each liquid. Fig. 7a summarizes the obtained results: for each liquid, from left to right, the orange bars represent the pendant drop measurements, the green bars show the Wilhelmy plate results, and the violet bars indicates the values reported in literature [20].

For water, a surface tension of 72.7 ± 0.5 mN/m was obtained using the pendant drop method, and 72.5 ± 0.02 mN/m using the Wilhelmy plate method. It is evident that the surface tension values obtained by both methods are coherent, with a very minor difference. Furthermore, both results closely align with the theoretical value (72.8 mN/m) [20]. This also applies to ethylene glycol and n-hexane. The pendant drop method yielded surface tension values of 45.9 ± 0.5 mN/m for ethylene glycol and 17 ± 0.1 mN/m for n-hexane, while the Wilhelmy plate method produced 48.2 ± 0.1 mN/m and 18.5 ± 0.1 mN/m respectively. These results are also in good agreement with the reported theoretical values in literature (48.0 mN/m and 18.4 mN/m respectively) [20]. These tests confirm the reliability of both adopted methods, as they yields consistent results with each other and with established literature values.

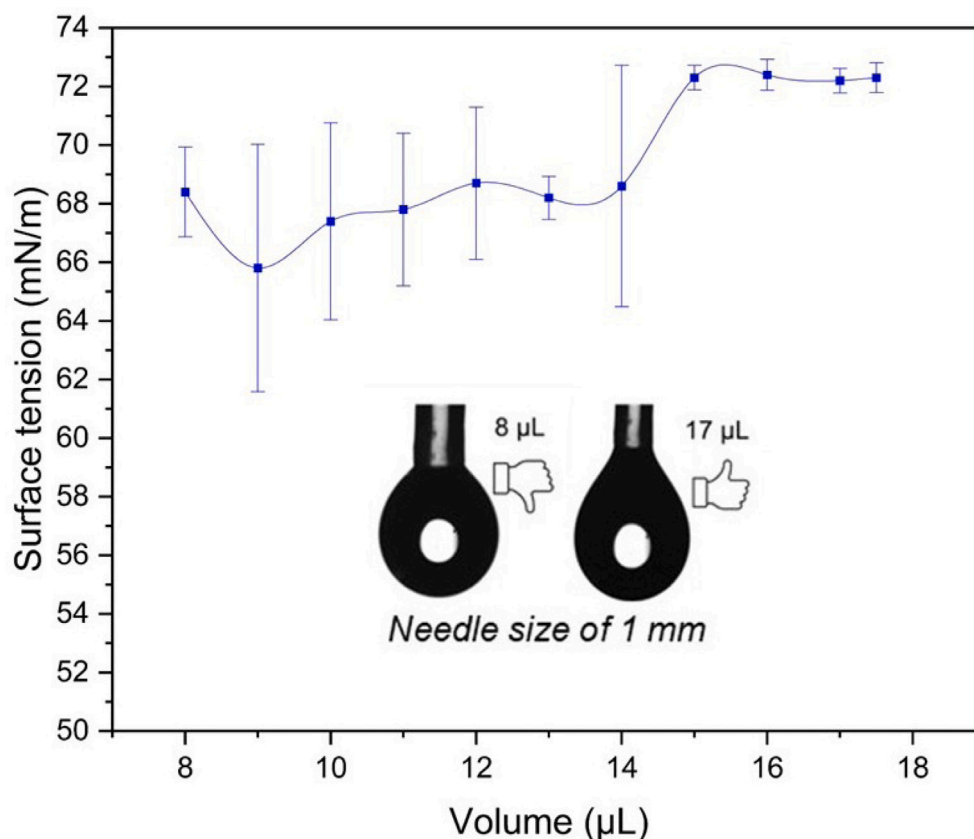


Fig. 5. The effect of pendant drop volume on the surface tension measurement of water at room temperature.







Liquid	Capillary length l_c	Needle Size			Theoretical γ_L (mN/m)
		1 mm	2 mm	3 mm	
		Experimental γ_L (mN/m)			
Water	2.71	72.3 ± 0.2 	72.7 ± 0.1 ✓ 	72.4 ± 0.4 	72.8
Ethylene Glycol	2.05	44.6 ± 0.5 	45.9 ± 0.55 ✓ 	39.8 ± 0.3 	48

Fig. 6. The effect of needle size selection on the surface tension measurement.

Next, Fig. 7b shows the surface tension of the selected liquid polymers measured using both methods. The surface tension measured using the pendant drop method for PEG 300, silicone oil, bio-resin, and paraffin oil were 44.8 ± 0.4 mN/m, 20.5 ± 0.7 mN/m, 45.7 ± 0.1 mN/m, and 31.7 ± 0.28 mN/m respectively. Similarly using the Wilhelmy plate method, the obtained results were 45.6 ± 0.09 mN/m, 21 ± 0.3 mN/m, 45.4 ± 0.2 mN/m, and 31.6 ± 0.1 mN/m respectively. Once again, the results demonstrated high accuracy and consistency between the two methods. Comparable values are also reported in literature, including 43.73 mN/m for PEG 300 [34], 46.7 ± 2 mN/m for the bio resin [35], 29.85 mN/m for paraffin oil [2,36], and 20.9 mN/m for silicone oil, as indicated in the manufacturer's data sheet. Moreover, the tested oils showed low surface tension values, as expected, due to the fact that they are totally dispersive.

3.3. Differential scanning calorimetry and thermogravimetric analysis results

Differential Scanning Calorimetry Results

Three tests were performed on each thermoplastic using the DSC method under a controlled nitrogen atmosphere with a heating rate of $10^\circ\text{C}/\text{min}$ to determine their melting temperatures. The results showed that polypropylene melts at $169.8 \pm 3.4^\circ\text{C}$ which falls in the $160\text{--}175^\circ\text{C}$ range reported in [37]. Similarly, PLA was found to melt at $169.7 \pm 0.7^\circ\text{C}$ which aligns well with the $165\text{--}167^\circ\text{C}$ temperatures reported by J. Hughes et al. in [38].

Thermogravimetric Analysis Results

Understanding the degradation temperatures of the selected polymers is essential to determine their respective thermal limits. Following

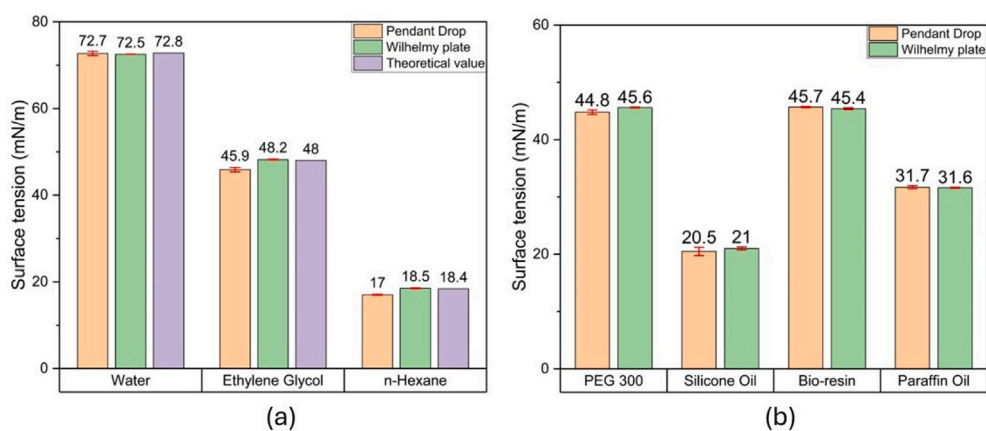


Fig. 7. (a) Test liquids surface tension measurement using the two adopted methods, (b) Liquid polymers surface tension measurement using the two adopted methods.

Table 2

Degradation temperature of various polymers at different isothermal steps.

Polymer	Sample weight loss (%)	Degradation temperature (°C)		Atmosphere conditions
		10 min isothermal steps	15 min isothermal steps	
PEG 300	5	160	160	Oxygen
Bio-resin	5	160	160	Oxygen
Silicone oil	5	300	300	Oxygen
Paraffin oil	5	220	200	Oxygen
PP	5	380	380	Nitrogen
PLA	5	300	290	Nitrogen

the programmed temperature cycle described in 2.2.5, and using a 5% weight loss as the criterion for the onset of degradation, the results are summarized in Table 2. The atmospheric conditions were selected according to the thermal behavior of the materials and the requirements of the surface tension measurements. For liquid polymers, the studied temperature range was relatively low (up to 100 °C) noting the rapid drop formation. Therefore, the surface tension measurements as a function of temperature were conducted in air. Oxygen was used for these liquids in this specific test, with the understanding that polymer degradation becomes more favorable at lower temperatures in an oxygen rich environment compared to air. In contrast, for thermoplastic polymers, higher temperatures and longer equilibration times were required for drop formation during the measurements. To eliminate any potential thermal or oxidative degradation under these conditions, nitrogen was selected as the purge gas for the tests.

It was observed that some polymers exhibit a higher degradation rate than the others, where extending the isothermal time from 10 to 15 min led to a reduction in their degradation temperatures. For instance the degradation temperatures of paraffin oil and PLA were 220 °C, and 300 °C respectively after 10-minute isothermal steps. However, when the isothermal time was increased to 15 min, their degradation temperatures decreased to 200 °C and 290 °C respectively. In contrast, for the remaining tested polymers, extending the isothermal step duration had no significant effect on their degradation temperatures.

To the best of our knowledge, no similar results have been reported in literature for the bio-based epoxy resin, silicone oil, and paraffin oil. In the case of silicone oil, the supplier's technical data sheet indicates a recommended service temperature of up to 200 °C. For PEG 300, A. Royer et al. reported that the degradation begins at 180 °C [39].

Regarding the thermoplastic polymers, previous studies have shown that polypropylene (PP) begins to lose weight at approximately 345 °C [40]. However, the temperature corresponding to a 5% weight loss was not specified. In the case of PLA, the degradation temperature at 5% weight loss reported in [41] is 315.4 °C, which is consistent with the results shown in Table 2.

The difference between our findings and those reported in literature can be attributed to the variation in the TGA testing methodology.

In our approach, the thermal program includes isothermal steps that interrupt the continuous heating cycle. In contrast, the temperature programs used in the referenced studies typically involve a continuous temperature ramp at a fixed heating rate, without interruptions. This methodological difference likely influences the observed degradation behavior. Moreover, those differences may be attributed to different polymer grades, as the source of polymers used in this study differs from that of the cited references.

3.4. Liquid and thermoplastic polymers surface tension versus temperature

Density determination versus temperature

Knowing that the pendant drop method is density dependent to calculate the surface tension, it was essential to start with determining the density of the selected polymers at each testing temperature before performing the surface tension test. As mentioned earlier in 2.2.3, the density of the polymer as a function of temperature was determined from the mass and volume measurements.

Fig. 8a shows the density results of PEG 300 in black squares, bio resin in red circles, silicone oil in blue triangles, and paraffin oil in green inverted triangles. Fig. 8b shows the density results of polypropylene in black squares and polylactic acid in red circles.

For thermoplastic polymers, the sessile drop technique was adopted for volume determination, and an analytical balance for mass measurement. While for liquid polymers, the pendant drop technique was firstly used to control the volume, then depositing the drops on a microscopic plate to measure their weight using an analytical balance. All polymers undergoes three tests at each temperature to get the corresponding density.

This technique was validated by measuring the densities of the liquid polymers at room temperature, then varying the temperature as shown in Fig. 8a. The measured densities of PEG 300, bio resin, silicone oil, and paraffin oil at room temperature were 1.23 ± 0.004 g/cm³, 1.14 ± 0.03 g/cm³, 0.95 ± 0.03 g/cm³, and 0.87 ± 0.002 g/cm³ respectively. These results are comparable with the values provided by the suppliers datasheet mentioned in 2.1.1. Similarly, the y-intercept of

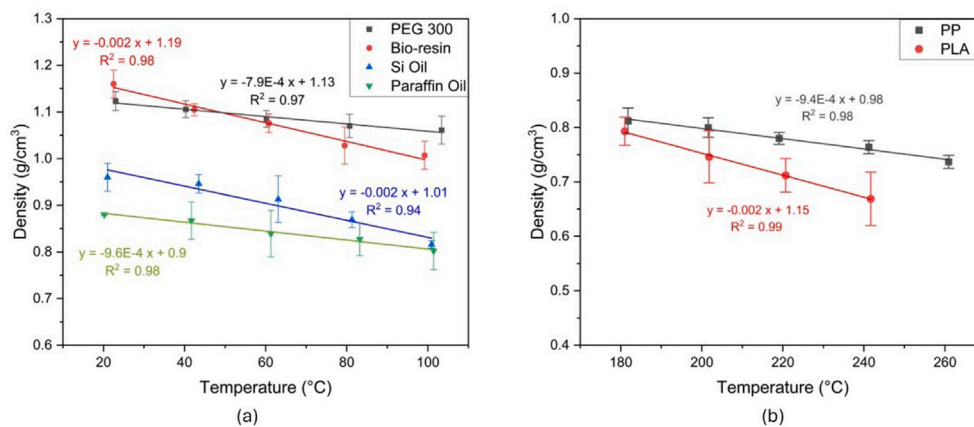


Fig. 8. (a) Liquid polymers densities versus temperature, (b) Thermoplastic polymers density versus temperature.

the linear fits shown in Fig. 8b (0.98 for PP, 1.15 for PLA) are coherent with the densities listed above in 2.1.1. For polypropylene, at 240 °C, the measured density was 0.764 ± 0.012 g/cm³ which is comparable to 0.732 g/cm³ reported in [42], as well as the results obtained by the PVT analysis over the covered thermal range reported in [43]. The densities were measured every 20 °C increase in temperature.

All polymers show a decrease in density with increasing temperature, which is expected as thermal expansion typically reduces density. Considering the high values of the coefficient of determination R^2 which is close to unity, the density of all the polymers decreases linearly as the temperature is increased. This linear decrease trend in density with polymers was also reported by Liang et al. in [44].

Surface tension determination versus temperature

After determining the required densities for the selected polymers, the surface tension of the liquid polymers were measured using the Wilhelmy plate (WP) and pendant drop methods (PD), as described earlier in Section 2.2. In the case of thermoplastic polymers, and due to the temperature limitation of the tensiometer apparatus (up to 120 °C), the surface tension of PP and PLA were determined exclusively using the pendant drop method. For each polymer and at each temperature, three tests were performed. Considering the parameters discussed in Section 3.1, the optimal needle size for each polymer was selected.

Fig. 9 presents four subplots (a–d), each illustrating how the surface tension of the tested polymers varies with temperature, as measured by the two adopted methods. Fig. 9a and b shows the surface tension variation against temperature for bio-resin and PEG 300 measured using the pendant drop method (red circles), and using the Wilhelmy plate method (black squares). Fig. 9c displays the results for silicone oil (black squares) and paraffin oil (blue triangles) obtained using the pendant drop method, and using the Wilhelmy plate (red circles for silicone oil and inverted green triangles for paraffin oil). A thorough review of the literature revealed few studies reporting similar methodologies for surface tension determination in such conditions, where Xu et al. studied Polyether ether ketone (PEEK) surface tension variation as function of temperature [45], while Yang et al. studied other thermoplastics [43]. The surface tension measurement obtained for each liquid and at various temperatures demonstrate a high degree of precision, as indicated by the low deviation observed across the three repeated tests. Furthermore, the close agreement between the surface tension curves derived from the two different methods confirms the high accuracy and repeatability of results, noting that the Wilhelmy plate method generally provide slightly higher surface tension values. Considering the high values of the R^2 coefficient, which is close to unity, the surface tension of the liquid polymers decreases linearly with increasing temperature. This trend is consistent with the Eötvös law [2,46].

Fig. 9d displays the results of three tests executed at each temperature for polypropylene (PP) represented by black squares, and

polylactic acid (PLA) shown as red circles, using the Pendant Drop (PD) method. Similar to the other liquid polymers, both thermoplastics exhibit a linear decrease in the surface tension with increasing temperature.

The surface tension of polypropylene at 200 °C was measured to be 29.54 ± 1.3 mN/m, which is in reasonable agreement with the value of 24.64 ± 0.02 mN/m reported in [47]. Moreover, it was reported that the surface tension of polypropylene at 240 °C is approximately 19 mN/m. This value was obtained using the pendant drop method with a needle having an outer diameter of 1.8 mm, an inner diameter of 1.3 mm, and a maximum drop volume of 7 μ L [42]. This reported value is somewhat lower than our measured result, which is 25.8 ± 1.6 mN/m at the same temperature. In our case, a needle with a diameter of 1 mm was used, and a drop volume ranged between 13 and 14 μ L. Although the needle dimensions are relatively similar, the discrepancy in surface tension values could be attributed to the difference in drop volume. As discussed in Section 3.1, it was observed that increasing the drop volume tends to increase the measured surface tension, bringing it closer to the known values of the tested liquids. This could explain the high value obtained in our measurement, as our drop volume is nearly double that used in [42].

As for PLA, the granules were dried overnight in a dehumidifier at 70°C prior to surface tension measurements as a function of temperature, following the procedure reported in [48].

Based on our linear fit, the y-intercept of PLA was found to be 71.9 mN/m. At a temperature of 25 °C, this corresponds to an estimated surface tension of approximately 67.7 mN/m. However, L. Jeantet et al. reported a significantly lower surface tension value of 43.9 ± 0.7 mN/m for PLA at room temperature [48]. This value was estimated using contact angle measurements formed between the probe liquids with known total, dispersive, and polar surface tensions, and a solid PLA substrate, noting that the PLA used in our study is different than that reported in [48].

Additionally, literature reports a surface tension of 30.81 mN/m for PLA at 180 °C, determined by numerical estimation [49], which is notably lower than our experimentally measured value of 43 ± 4 mN/m at the same temperature.

The difference between our measured values and those reported in the literature can be justified by the different methodologies employed. While our measurements were obtained using the pendant drop method, other studies have used alternative techniques. Additionally, the inherent complexity of handling PLA, along with the high variability observed in both the measured density (Fig. 8b) and surface tension (Fig. 9d), further contributes to the observed deviation.

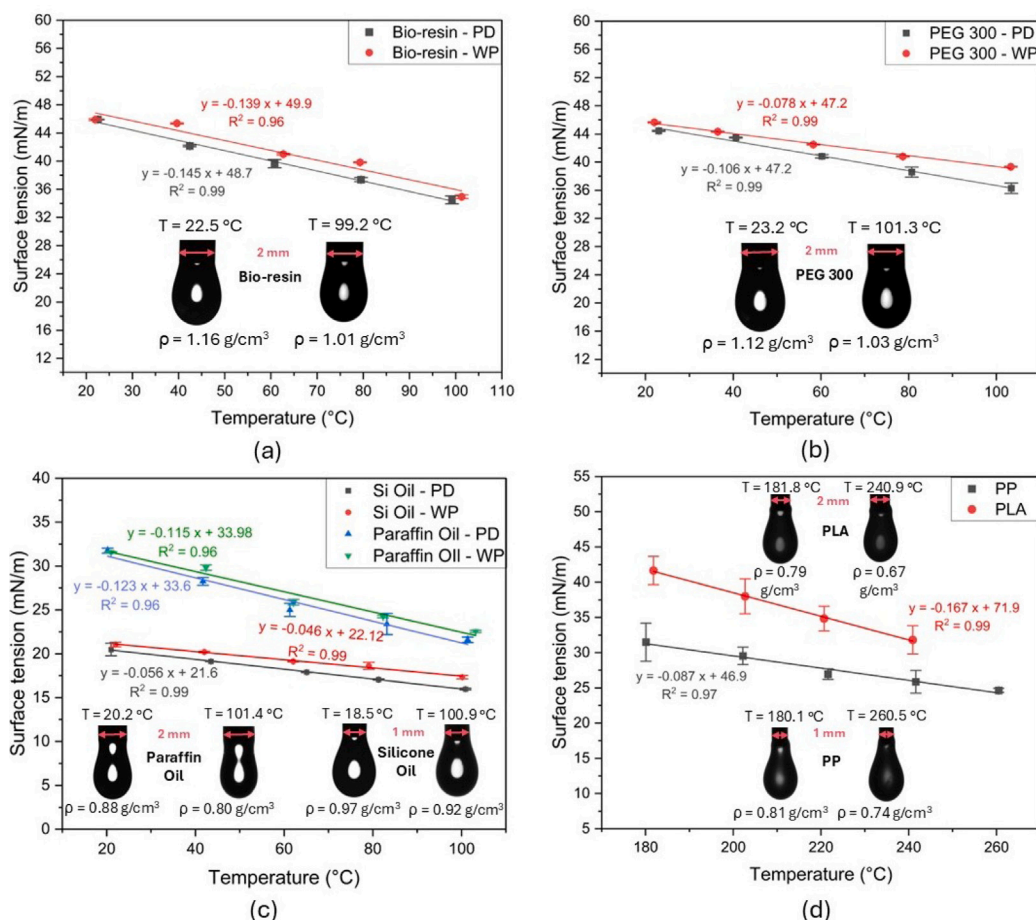


Fig. 9. Surface tension as a function of temperature for the selected polymers using the Wilhelmy Plate (WP) and Pendant Drop (PD) methods: (a) Bio resin, (b) PEG 300, (c) Silicone oil and Paraffin oil, (d) PP and PLA.

3.5. Liquid polar and dispersive component determination

The polar and dispersive components of the tested polymers were determined using Eqs. (2) and (3). To develop an experimental protocol that could be later applied with temperature, it was first essential to begin with a liquid with well-established properties. For this purpose, ethylene glycol was selected.

Traditionally, n-hexane is commonly used for interfacial tension measurements. However, due to its limitation at elevated temperatures, it was necessary to identify an alternative liquid that is purely dispersive and has a density lower than that of the tested polymers. Therefore, silicone oil and paraffin oil were selected as potential replacements.

To confirm that the two liquids are entirely dispersive, each was subjected to three interfacial tension measurements using n-hexane by applying the Wilhelmy plate method. The pendant drop method was not applicable because silicone oil, paraffin oil, and n-hexane are totally wetting liquids, preventing stable drop formation. For silicone oil, the average polar component was calculated to be 0.04 ± 0.08 mN/m, while the dispersive component was 20.46 ± 0.08 mN/m. In the case of paraffin oil, a value of 0 ± 0.35 mN/m for polar component and 31.6 ± 0.35 mN/m were determined. These results confirm that both liquids meet the requirement of the Owens and Wendt equation for use as purely dispersive reference liquids. Thus, $\gamma_L = \gamma_L^d$ at the studied temperature.

The results of the three interfacial tension tests between ethylene glycol with n-hexane, silicone oil and paraffin oil are presented in Fig. 10a. Using both Wilhelmy plate and pendant drop methods, Fig. 10b displays the polar and dispersive components of ethylene glycol calculated based on the interfacial tension values shown in Fig. 10a.

First, when comparing the results obtained using the two methods, it is obvious that both approaches are reliable. For instance, the polar and dispersive results of ethylene glycol with n-hexane show minimal deviation when between the two methods. This observation also holds true for the results obtained with the two other low-density liquids.

The consistency of the polar and dispersive components across the low-density liquids is noteworthy. The values obtained show a high degree of agreement with the theoretical values previously reported in Table 1. Therefore, the use of silicone oil and paraffin oil as a replacement of n-hexane in interfacial tension tests appears to be a promising and valid approach.

After confirming the compatibility of silicone oil and paraffin oil with the interfacial tension test, PEG 300 and the bio-resin were subsequently tested to determine their polar and dispersive components, following the same procedure used for ethylene glycol.

Fig. 11a–c present the averages of three interfacial tension measurements for the bio-resin and PEG 300, respectively, using the three low-density liquids and both the Wilhelmy plate and pendant drop method. Fig. 11c–d display the polar and dispersive components of PEG 300 and bio-resin, calculated from the previously three interfacial tension tests using both methods as well.

Starting with the measured interfacial tension results, it is observed that the lowest values occur when the polymers are tested with n-hexane as a low-density liquid. In contrast, higher interfacial tension values were determined when silicone oil and paraffin oil were used. As for the observations with ethylene glycol, the results obtained using the two methods are comparable. However, in the case of paraffin oil, the interfacial tension values measured by the pendant drop method are slightly lower than those obtained with the Wilhelmy plate method.

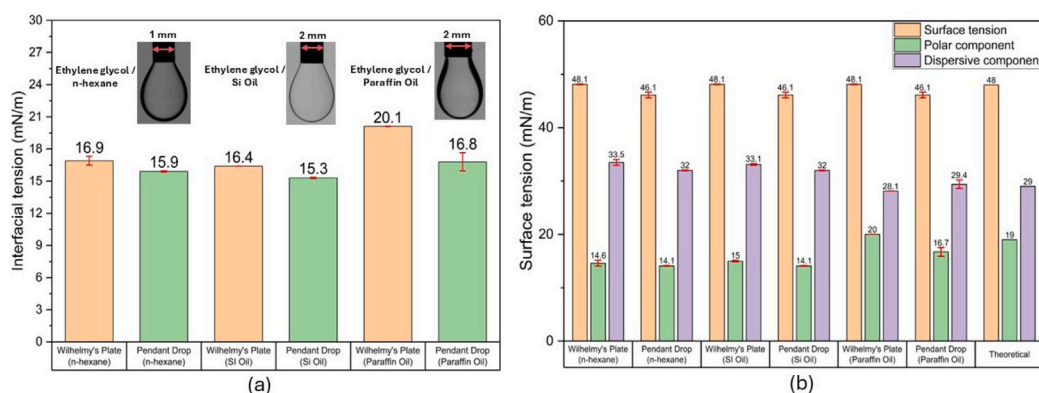


Fig. 10. (a) Interfacial tension results using the three low density liquids using the WP and PD method, (b) Polar and dispersive components of ethylene glycol determined using the results of the interfacial tension tests.

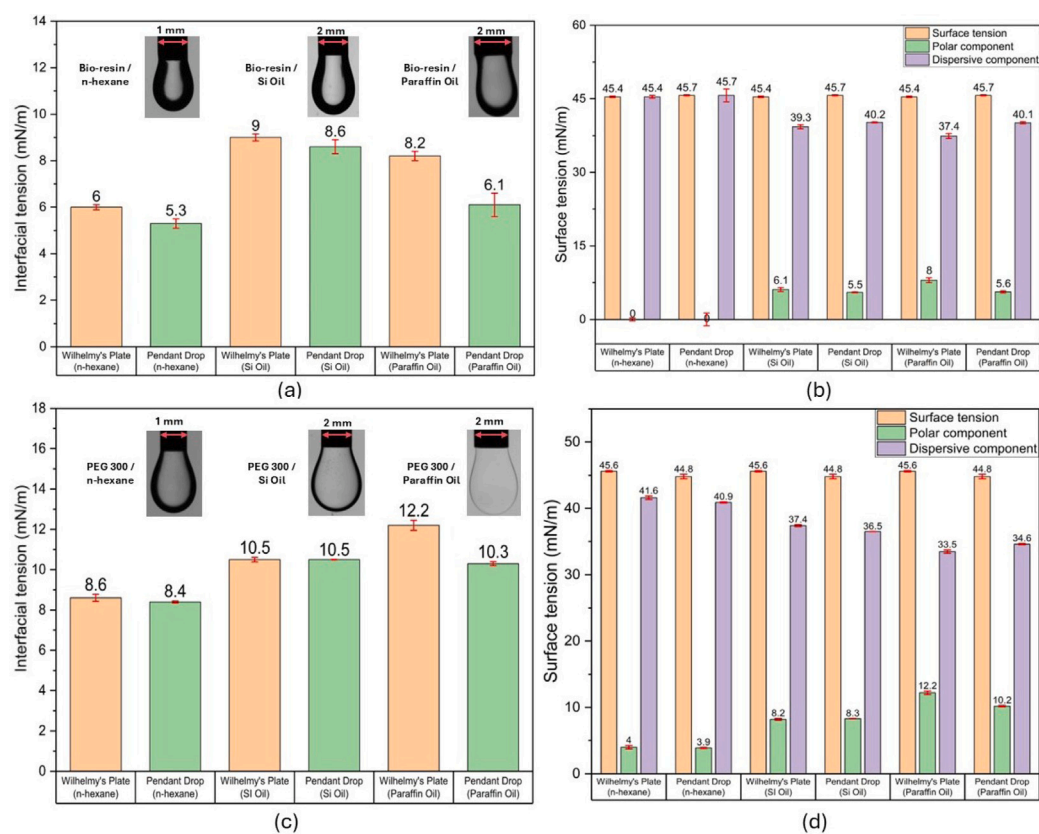


Fig. 11. (a) Bio-resin interfacial tension test results with the three low density liquids using WP and PD methods, (b) Polar and Dispersive component determination for the Bio-resin, (c) PEG 300 interfacial tension test results with the three low density liquids using the WP and PD methods, (d) Polar and Dispersive component determination for PEG 300.

This discrepancy may be attributed to the asymmetric pendant drop shape formed inside the cuvette filled with paraffin oil. The pendant drop formed between the bio-resin and paraffin oil shown in Fig. 11a, clearly illustrates the asymmetrical drop phenomenon. The underlying cause of this asymmetry remains to be investigated.

After calculating the polar and dispersive components of the tested polymers, it is obvious from Fig. 11b–d that differences exist among the results. The values obtained with silicone oil and paraffin oil as low-density liquids are comparable to each other and demonstrate greater consistency than those obtained using n-hexane as the low-density liquid.

To better understand the differences observed in interfacial tension and in the polar and dispersive components, a solubility test was

performed on PEG 300 using the pendant drop method. In this test, a PEG 300 pendant drop was formed inside the cuvette containing the selected low-density liquids. Following the initiation of the measurement, the drop shapes evolution was monitored and recorded at 0, 60, and 120 min.

Fig. 12 presents the results of this test. For PEG 300 suspended in silicone oil and paraffin oil, no changes in the shape or morphology of the drops were observed over time. In contrast, for PEG 300 with n-hexane, dark dots began to appear on the drop surface after 60 min and became more noticeable after 120 min. These observations indicate a degree of solubility between n-hexane and PEG 300. Similar phenomena regarding the solubility of alkanes in polymers have been previously reported in literature [50,51].

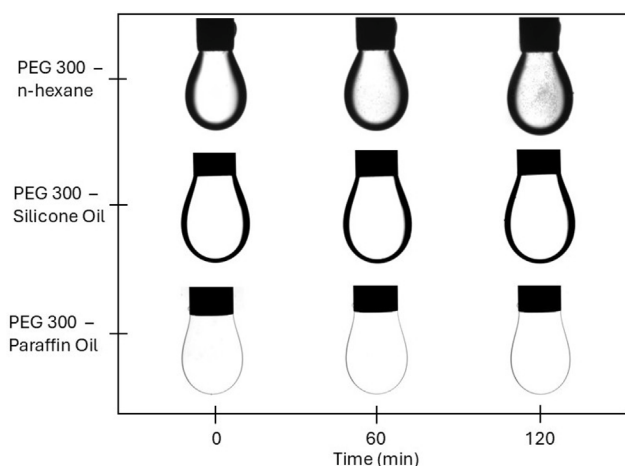


Fig. 12. Solubility results of PEG 300 in n-hexane, silicone oil, and paraffin oil as a function of time.

Due to the solubility of n-hexane in PEG 300, the interface between the two liquids becomes less distinct, which can lead to an inaccurate estimation of the interfacial tension. Furthermore, Villers and Platten [52] reported that, at a constant temperature, an increase in the degree of solubility corresponds to a decrease in interfacial tension. This observation supports our results and provides an explanation for discrepancy in the interfacial tension values in Fig. 11a–c and especially the lower interfacial tension values obtained when PEG 300 and the bio-resin were tested with n-hexane.

Therefore, it is demonstrated that silicone oil and paraffin oil are suitable and reliable alternatives to n-hexane, enabling interfacial tension measurements with liquid and thermoplastic polymers at elevated temperatures, which will be conducted in forthcoming studies.

4. Conclusion

The present study details a comprehensive experimental approach for the accurate estimation of surface tension and its polar and dispersive components in polymers, that is relevant for estimating fiber–matrix adhesion, which is a key factor governing the mechanical performance of fiber-reinforced thermoplastic composites. Reliable and validated methodologies were developed using both the Wilhelmy plate and pendant drop techniques to measure surface tension as a function of temperature. The temperature dependent surface tension of PEG 300, bio-resin, silicone oil, and paraffin oil were successfully estimated using both methods, while the surface tension of polypropylene and polylactic acid were determined using the pendant drop method. Understanding the variation of this parameter with temperature provides critical insight into optimizing fiber wetting behavior, thereby enabling more precise control over interfacial interactions in composite processing. These key findings form the basis of ongoing works, which include fiber wetting studies with these liquid and thermoplastic polymers, to determine static and dynamic contact angles with different fibers. In addition, interfacial shear strength (IFSS) tests are planned to provide a more comprehensive assessment of the mechanical performance of the fiber–matrix interface.

Furthermore, a systematic procedure was developed to determine the polar and dispersive components of polymer surface tension using both methods. A key advancement was the identification of silicone oil and paraffin oil as suitable replacements for n-hexane, thereby enabling these methods to be reliably extended to elevated temperatures. This development not only provides deeper insights into polymer surface behavior but also establishes an effective framework for optimizing adhesion and enhancing fiber–matrix interfacial performance in

composite manufacturing. Building on these key results, the methodology can now be applied to investigate advanced surface properties of molten polymers.

CRediT authorship contribution statement

Rami Alawar: Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Pierre-Jacques Liotier:** Writing – review & editing, Visualization, Validation, Supervision, Methodology, Investigation, Conceptualization. **Romain Ravel:** Writing – review & editing, Methodology, Investigation, Conceptualization. **Monica Francesca Pucci:** Writing – review & editing, Visualization, Validation, Supervision, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

The authors acknowledge the financial support of the French National Research Agency (ANR) under grant number ANR-23-CE51-0005 (DUINTACOS project).

Data availability

Data will be made available on request.

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