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Optimizing parameters to improve PDMS surface wettability and the thermal conductivity analysis

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ABSTRACT

Due to its remarkable characteristics, Polydimethylsiloxane (PDMS) is widely used in microfluidic devices. However, despite its advantageous physical and chemical properties, its hydrophobic nature poses a challenge when pumping aqueous solutions through microchannels using only capillary forces. Various methods have been proposed to increase the hydrophilicity of PDMS; however, many struggle with hydrophobic recovery within a short time, whereas most commercial devices require long-term stability for storage and distribution. Incorporating surfactants into PDMS has become a promising technique for reducing hydrophobicity and regulating its recovery over time. However, selecting the right surfactant requires a thorough evaluation of its effectiveness, stability, and long-term durability in maintaining hydrophilicity. In this study, three non-ionic surfactants with different critical micelle concentrations and chemical compositions were compared: Triton X-100, Brij L4 (BL4), and Polyethylene Oxide (PEO). For this purpose, different surfactant concentrations, curing temperatures, and types of surfactants were compared. Short- and long-term experiments were conducted, in which deionized water droplets were placed on the surface of PDMS mixed with surfactants to assess wettability. Additionally, the influence of surfactants on thermal conductivity was analysed, using a Hot Disk 5501 sensor. The Taguchi method results identified the optimal sample as 2.5 % PEO cured at 80 °C, which achieved a contact angle of 12.8° immediately after curing and maintained superior wettability both at 0 h and after 3 weeks of curing. For the initial thermal conductivity (0 h), the optimal sample was 0.5 % TX-100 at 80 °C, and after 3 weeks, BL4 2.5 % at 25 °C. To identify the best overall sample considering both tests, the Grey Relational Analysis method was applied. Additionally, an ANOVA statistical analysis was performed to evaluate the percentage of influence of each parameter, both in the Taguchi method and in individual tests, as well as in the Grey Relational Analysis, which combined both methods.

1. Introduction

Polydimethylsiloxane (PDMS) is a polymer widely used in various technological applications due to its unique properties, such as flexibility, transparency, biocompatibility, and ease of manufacture [1–4]. However, the intrinsic hydrophobicity of PDMS poses challenges in microfluidic applications. This hydrophobic nature hinders the transfer of aqueous solutions, particularly evident in processes such as microcontact printing, where the hydrophobic surface of PDMS obstructs fluid flow. Furthermore, pressure-driven liquid processing equipment experiences a significant drop in internal pressure in the microchannels surrounding the hydrophobic PDMS surface, exacerbating the issue. Consequently, various surface treatment methods have been explored to reduce this challenge and improve the wettability of PDMS surfaces

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[5,6].

Among these methods, gas-phase processing techniques such as oxygen plasma [7–9], UV/ozone treatment [9–11], UV irradiation [12] and electrical discharge [13] have been widely investigated. Although these methods initially produce hydrophilic surfaces, their effectiveness is limited by the short lifetime of the hydrophilic modification due to the diffusion of low molecular weight chains [14–16]. Chemical processing methods, including chemical vapour deposition (CVD), layer-by-layer deposition (LBL), sol-gel coatings and salinization, have also been used to improve the hydrophilicity of PDMS [17–20]. However, these methods often involve complex protocols, multi-step procedures and high-cost facilities, posing significant challenges, especially for inexperienced users. Another commonly used approach is the incorporation of nanomaterials, but concerns over stability, toxicity, diffusion and the migration of nanoparticles, especially toxic ones, make this method less desirable [9].

Currently, the use of surfactants has emerged as a promising solution to address the hydrophobicity of PDMS surfaces [21–25]. Surfactants, as wetting agents, offer a promising approach to reducing fluidic resistance in microchannels by lowering surface tension and facilitating liquid spreading. This method avoids specialized equipment and complex procedures, making it accessible even to beginners. Surfactant-modified PDMS has demonstrated significant potential in various microfluidic applications, such as biomolecule separation, blood cell sorting, immunological assays, and drug delivery. The addition of surfactant to PDMS has demonstrated biocompatibility and stability, making it a viable option for various industrial and research applications [26]. Among the various studies that have investigated the incorporation of surfactants into PDMS, several have demonstrated remarkable innovation and impact. O'Brien and Paranjape [27] focused on modeling dynamic surface tension and fitting contact angles over time; however, they did not examine the type of surfactant, concentration, and long-term effects. Condelipes et al. [28] incorporated a single ethylene oxide block copolymer at 1 wt% and demonstrated its application in capillary immunoassays, nevertheless without performing any statistical optimization or analyzing thermal properties. Seo and Lee [29] used 3 wt% Triton X-100, and they observed a rapid decrease in water contact angle and tracked its recovery over 30 days, yet they have only tested a single concentration and did not assess thermal conductivity. Park et al. [30] embedded non-ionic surfactants into PDMS and verified a reduction in the initial contact angle from 153° to 134°; however, they did not explore different surfactant types or concentrations, nor did they perform any thermal analysis. Lastly, Lee and Lee [31] have employed Silwet L-77 in moulds for hot embossing, and they have established a wettability correlation with high replication fidelity. Even so, they did not include aging tests or thermal analysis.

In the present study, the authors aimed to address some of the limitations reported in previous works by evaluating the effects of three different surfactants on PDMS: Polyethylene Oxide (PEO), Brij L4 (BL4) and Triton X-100 (TX-100). The primary objective was to improve the wettability of the PDMS, facilitating fluid flow via capillarity in microchannels without the need for pumps or other forced movement devices. In addition, the thermal conductivity of the PDMS-surfactant mixture was analysed to determine whether increased hydrophobicity enhanced hydrophilicity.

The adopted methodology involved preparing PDMS samples with varying concentrations of each surfactant and different curing temperatures. An orthogonal arrangement using the Taguchi method was used to select parameter combinations and sample levels. Once the arrangement had been defined, the samples were made, and then the detailed wettability and thermal conductivity tests were carried out. Since the Taguchi method only identifies the ideal sample for each test individually, Grey's Relational Analysis was employed to combine both tests allowing for a more comprehensive evaluation. The final ideal sample was determined by considering the percentage influence of each test on the overall result.

Wettability improvements were evaluated using contact angle measurements, where a lower contact angle indicates higher surface hydrophilicity. Thermal conductivity was measured using standardized techniques to assess the impact of surfactants on PDMS's heat dissipation capacity.

The findings of this study have significant implications for developing more efficient microfluidic devices. Increasing the hydrophilicity of PDMS led to substantial improvements in capillary flow tests, with potential applications in areas such as medical diagnostics, small-scale chemical synthesis and cell manipulation. Although the improvement in thermal conductivity was modest, it opens new possibilities for using PDMS in devices requiring precise thermal control, such as cooling systems for microchips or other electronics that demand both good wettability and thermal conductivity.

This study provides valuable insights into the modification of PDMS by identifying suitable surfactants to enhance its surface and functional properties. Additionally, by incorporating multi-response optimization, long-term stability evaluation, and dual characterization of wettability and thermal conductivity, this work represents a significant advance in PDMS surface engineering for microfluidic and thermal applications, marking an important step toward optimizing materials for more advanced uses in microtechnology and nanotechnology.

2. Materials and methods

For this study, non-ionic surfactants were chosen because these organic compounds are amphiphilic, which means they can mix with both polar and nonpolar substances. Each surfactant used had distinct chemical names and Hydrophilic-Lipophilic Balance (HLB) values, which measure a surfactant's relative affinity for water or oil. The surfactants used were: Brij L4 (SIGMA-ALDRICH), Triton X-100 (SIGMA-ALDRICH) and Polyethylene Oxide (Polysciences Europe GmbH).

2.1. Taguchi method

The Taguchi method [27] is a statistical approach designed to optimize processes and projects by reducing variability and enhancing performance through controlled experiments. This method is based on experiment planning procedures to identify the most influential factors in each system and determine its optimal configurations, aiming to improve product or process quality and robustness.

In the Taguchi method, the signal-to-noise ratio serves as a measure of robustness. It helps identify control factors that reduce variability in a product or process by minimizing the impact of uncontrollable factors (noise factors). Control factors are parameters that can be adjusted, while noise factors cannot be controlled during the production or use of the product but can be managed during experimentation.

In the experiment, two signal-to-noise ratios were applied: one for wettability, where lower values are preferred since the goal is to minimize wettability, and another for thermal conductivity, where higher values are preferable to maximize conductivity. Initially, the study focused on improving the surface wettability of PDMS, with parameters and levels selected based on relevant properties from the literature [28–30]. Author Seo et al. [29] said that adding surfactants to the surface and bulk of PDMS was one way to change its wettability. The parameters studied when adding surfactants include the type of surfactant, concentration percentage, and curing temperature of the PDMS-surfactant mixture. Three basic levels for each parameter were defined

Table 1
Parameters and levels Taguchi.

Parameter	Level 1	Level 2	Level 3
Surfactant concentration (%)	0.5 %	1 %	2.5 %
Curing temperature (°C)	25 °C	80 °C	120 °C
Type of surfactant	Brij L4	PEO	Triton X-100

to select the appropriate Taguchi array, as shown in Table 1.

Knowing the number of parameters and levels, the appropriate orthogonal array was selected. In this case, with 3 parameters at 3 different levels, the proper Taguchi's orthogonal array is L_9, as shown in Table 2.

This study aims to evaluate the decrease in the contact angle on the PDMS surface and the increase in thermal conductivity. Upon completing the wettability and conductivity tests, the goal is to identify the optimal combination of parameters that yields both lower wettability and higher conductivity.

2.2. ANOVA analysis

Analysis of variance (ANOVA) is a statistical method used to compare different levels of one or more test variables, allowing for the determination of whether the means of three or more groups (levels) are significantly different. Variance, which measures dispersion, calculates the extent of deviation of the data from the sample mean. One of the main objectives of ANOVA is to identify whether there are significant differences between the distributions of results in a sample involving three or more groups [31].

To perform the analysis of variance, an ANOVA table is created to calculate the effects of each factor included in the analysis. These factors are: Sequential sums of squares indicate the variation attributed to the different components of the model; Degrees of freedom (DF) represent the amount of information available in the model; Mean squares are obtained by dividing the sum of squares by the degrees of freedom and represent the variation between sample means; The F-value is a test statistic used to determine whether a control factor in the model is related to the response and to measure the extent of its influence; The *P*-value is the probability of obtaining a value of the test statistic equal to or greater than the observed one. Generally, a P-value less than 0.05 is used as a criterion to reject the null hypothesis, indicating that an extreme value for the test statistic would occur less than 5 % of the time if there were no significant differences [32–34].

2.3. Grey relational analysis

The Grey Relational Analysis technique uses a specific relational grade to determine the proximity between tests. This method normalizes experimental results, including wettability and thermal conductivity, on a scale from zero to one according to their relevance. Grey's relational coefficient is then calculated from this normalized data, showing the relationship between the desired and actual results. The Grey relational grade is then achieved by averaging these coefficients, reflecting the overall assessment of the various responses in the process. The procedure, also known as "GRA" (Grey Relational Analysis), follows a sequence of defined steps [34–36].

Table 2
Taguchi L_9 orthogonal array with parameters and levels.

Arrays	Surfactant concentration %	Curing temperature (°C)	Type of surfactant
1	0.5 %	25 °C	BL4
2	0.5 %	80 °C	PEO
3	0.5 %	120 °C	TX-100
4	1 %	25 °C	PEO
5	1 %	80 °C	TX-100
6	1 %	120 °C	BL4
7	2.5 %	25 °C	TX-100
8	2.5 %	80 °C	BL4
9	2.5 %	120 °C	PEO

2.4. Sample preparation

2.4.1. Bulk PDMS modification method

Firstly, the PDMS (Sylgard 184TM, Silicone Elastomer Base, USA) was prepared by mixing a base and a curing agent in a 10:1 weight mixing ratio for 5 min in a slow spiral motion in the same direction. The mixture was then degassed for 10 min. Following this, the Brij L4, PEO and Triton X-100 surfactants were added in proportions of 0.5, 1 and 2.5 wt%, and this mixture was also degassed for 10 min [37]. The surfactant/PDMS mixture (s-PDMS) was then poured into a mould to standardize the sample's diameter, thickness and surface roughness. It was then put back into the mould for degassing again and, finally, the samples were cured at temperatures of 25 °C, 80 °C and 120 °C. The entire process is illustrated in Fig. 1.

To meet the manufacturer's specifications for the Hot Disk 5501 F1 sensor, used for the thermal conductivity test, the samples were made in a mould with a thickness (h) of 1.1 cm and a diameter (D) of 3 cm. It consisted of a machined aluminium part with four holes and four gaps to contain excess material from each hole, preventing material placed in one hole from coming into contact with the material in another hole, which usually happens when bubbles are removed, as the material on the surface expands and tends to go sideways. For the base of the mould, a rectangle of smooth glass is placed between the part with the holes and two small plates, which are secured by screws, washers, and nuts.

2.4.2. Microchannel fabrication for capillary studies

The production of polymeric microfluidic devices for open capillary assays started with designing a standard model using Inventor software (version 27.0). Four different types of channels were designed: (1) a straight rectangular channel with a length of 42 mm and a width of 1 mm (Fig. 2A); (2) a spiral-shaped channel with a length of 42 mm and a width of 1 mm (Fig. 2B); (3) a main channel with a width of 1 mm that splits into two equal branches (Fig. 2C); and (4) a channel with bifurcation-confluence geometry (Fig. 2D). All channels had a depth of

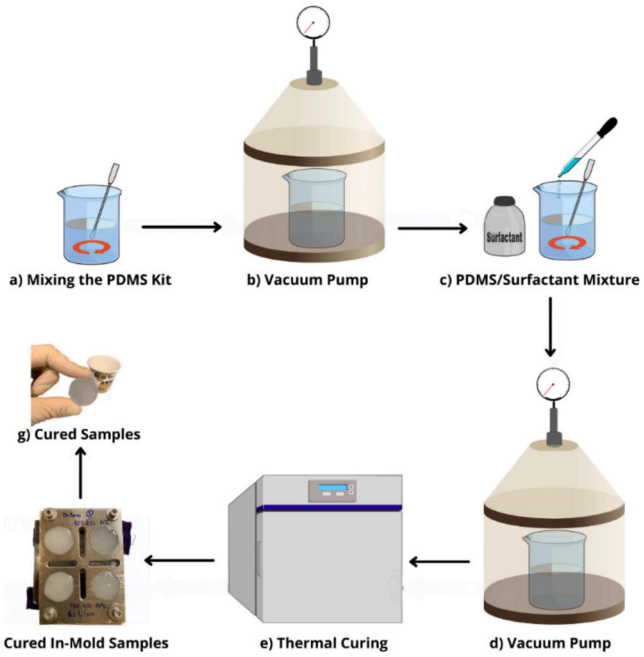

Fig. 1. Schematic of the manufacturing process for PDMS specimens with added surfactant, by bulk mixing. Where (a) shows the PDMS kit (PDMS and curing agent), (b) bubble's removal from PDMS kit mix, (c) mix PDMS kit with surfactants (Brij L4, PEO, Triton X-100) at 0.5, 1 and 2.5 wt%, (d) removing bubble from the PDMS kit mixture with surfactants (already poured into the mould), (e) curing the samples at 25 °C, 80 °C and 120 °C, (f) cured samples in the mould and (g) final cured and demoulded sample.

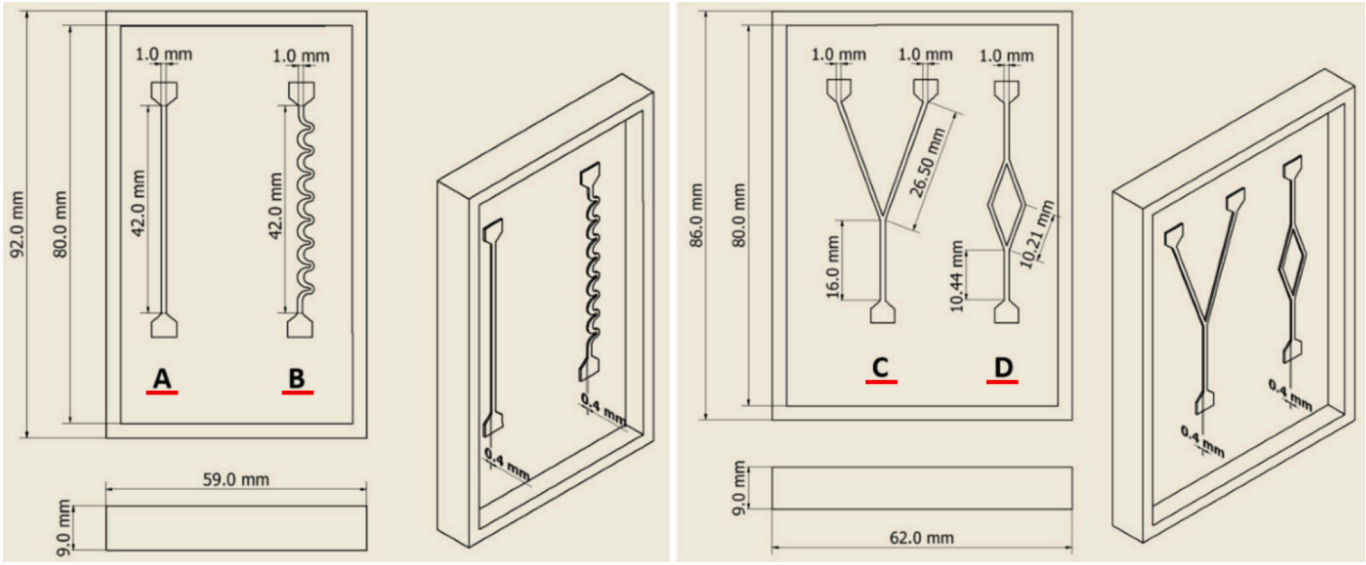

Fig. 2. Designs and dimensions of the different moulds for the capillary microchannels. Four channels were drawn: (A) a straight rectangular channel; (B) a channel with spiral-shaped geometry; (C) a channel with a main channel that bifurcates into two equal branch channels; and (D) a channel with bifurcation-confluence geometry [28].

0.4 mm [28].

To create the microchannel moulds, the drawings were converted into .STL format to be exported to the 3D printer pre-processing software, CHITUBOX V1.9.5 (Shen-zhen, China, CBD-Tech). The used printer was the Anycubic Photon D2 (Anycubic Company, China), a 3D printer based on Digital Light Processing. A specific translucent photosensitive resin (Standard Resin Clear, Anycubic Company, China) was used in the printing process, and the high-resolution parameters were defined. Post to the printing process, the remaining uncured resin was washed with 99.9 % isopropyl alcohol (EQM Soluciones Quimicas, Madrid, Spain), for 10 min, in the Anycubic Wash & Cure Plus Machine (Anycubic Company, China), to remove any excess of resin that remained on the microchannel surface mould. Afterwards, it was subjected to a UV curing session lasting 2 h in the Anycubic Wash & Cure Plus Machine (Anycubic Company, China). The UV light further cured the resin, making the moulds more rigid and improving their structural integrity. Lastly, the mould was exposed to a 24 h thermal treatment, at 80 °C, in an oven.

2.5. Contact angle measurements

Static contact angle measurements were performed using the sessile drop technique to assess the wettability of the PDMS samples. Both modified and pure (control) PDMS samples were carefully cleaned with damp paper. Using a 100 μL micropipette, a 10 μL volume of deionized water was carefully dispensed onto the sample surfaces. The water contact angle (WCA) was determined using a goniometer connected to an optical microscope and a computer with contact angle (CA) measurement software, including SCA202 software for OCA and PCA. To ensure accurate measurements, the lighting conditions, focus, baseline, and contour line were adjusted. The image of the drop on the PDMS sample was captured immediately after sample preparation (0 h) and at subsequent intervals of 1 week, 2 weeks, and 3 weeks. During each test run, WCA was measured every minute for 10 min, obtaining 10 measurements over 10 min at three different points in the sample, to have a detailed analysis of the changes in the hydrophobic properties of PDMS over time, as shown in Fig. 3.

2.6. Thermal conductivity measurements

The thermal conductivity (κ) of a material is the property that defines its ability to conduct heat. It quantifies the amount of heat (Q) that passes through a material of thickness L and area A in each time interval (t), when there is a temperature difference (ΔT) between the opposite sides of the material. The Fourier equation for heat conduction in one dimension provides the relationship [38].

$$Q = -\kappa A \frac{\Delta T}{L} t \quad (1)$$

The unit of measurement for thermal conductivity in the

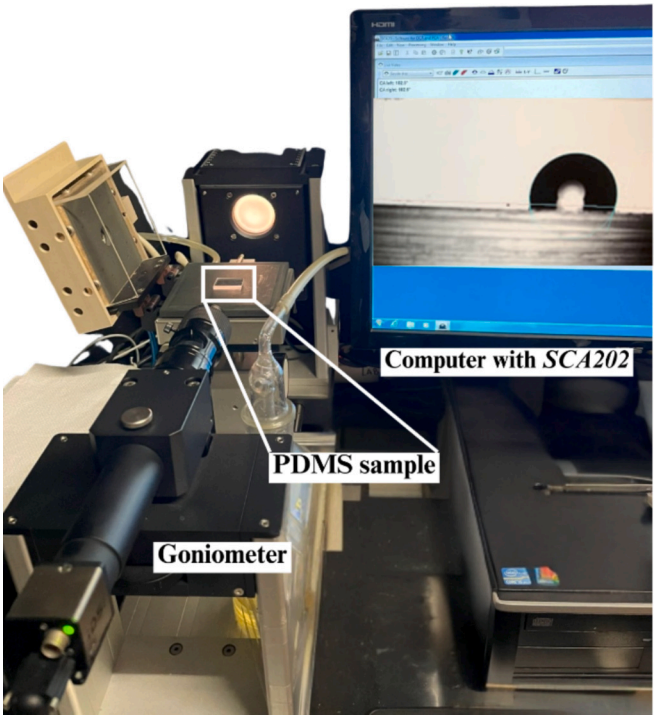

Fig. 3. Measurement of the contact angle using an optical microscope (goniometer), connected to a computer with the CA measurement software, SCA202. Images captured by the goniometer with a 10 μL drop of distilled water on samples.

International System (SI) is the watt per meter-kelvin (W/mK). The method employed in this work was the Hot Disk method.

2.6.1. Hot disk TPS 2500S

Transient plane source (TPS) equipment provides data on thermal properties, including thermal conductivity, thermal diffusivity and specific heat per unit volume. These measurements follow the ISO 22007-2 standards as specified by the manufacturer [39]. The Hot Disk Thermal Constant Analyser is a method that employs a flat heated sensor. According to the TPS 2500S equipment manual, it can measure thermal conductivity values ranging from 0.005 to 1800 W/mK, with reproducibility commonly exceeding 1 % and accuracy exceeding 5 %. It is compatible with both standard isotropic measuring modules and additional components [40–43].

2.6.2. Temperature and heat flux sensor

Hot Disk sensors are composed of thin polyimide films (Kapton®) with thicknesses ranging from 12.7 μm to 25 μm, designed for use at cryogenic temperatures up to 300 °C. The total sensor thickness varies from 60 μm to 80 μm. The sensor used in this study, Hot Disk 5501 F1, has an external radius, r_{Kapton}, of 10 mm while its internal radius, r_{Hot}, of 6.4 mm in a spiral. Four electrical connections are provided for the sensor's double spiral: two for conducting the heating current and two for controlling the voltage drop. The TPS 2500S system was adapted to monitor resistance variations during the transient heating of the sample.

According to the manufacturer's specifications for the Hot Disk 5501 F1 sensor, the sample thickness must satisfy the condition $r \leq h \leq 2r$, and for the sample diameter, $4r \leq D \leq 6r$, where r refers to the r_{Kapton} radius of the sensor.

For thermal property measurements (Fig. 4), the Hot Disc sensor emits a controlled heat pulse for a predetermined period. The amplitude, penetration depth and duration of this pulse depend on the response of the material used as the test sample. To establish a reference, a pure PDMS sample was used and adjusted to the initial conditions, thus serving as a reference for further analyses. To ensure the accuracy of the measured values, the results were compared with data from the literature on the thermal conductivity of pure PDMS, as well as with data provided by material suppliers.

Each test series consisted of five consecutive measurements, each lasting 40 s, with 40 mW of power applied to the sample. There was a 2-min interval between measurements to allow the temperature to stabilize. The sample temperature was monitored using a PT100 sensor. Standardized settings, including time correction, no offset compensation, and standard heat capacity, were applied. Insulation conductivity values of 0.036 W/mK and diffusivity of 0.69 mm^2/s were also considered.

3. Results and discussion

This chapter presents and discusses the results of contact angle and thermal conductivity measurements for PDMS samples with added surfactants, as well as pure PDMS samples. The evaluation was carried out using the Taguchi method, focusing on two main aspects: first, the signal-to-noise (S/N) ratio, which helps determine the level of variation for each parameter, and second, analysis of variance (ANOVA), used to assess the influence of each parameter. Taguchi, ANOVA and Grey analysis were applied to the results from the tests in the initial phase (0 h) and after 3 weeks, to assess both the immediate and medium/long-term behavior of the samples with surfactant. All reported uncertainties are expanded uncertainties with a 95 % confidence level (k = 2), unless otherwise specified.

3.1. Wettability test

3.1.1. Taguchi method results

As the aim is to obtain the lowest possible surface wettability (lowest contact angle), the S/N ratio for this parameter is "lower is better", as it allows the value of the contact angle to be minimized. With the values obtained from the wettability tests (Fig. 5) and applying the Taguchi method, it was possible to determine the signal-to-noise ratios for the nine samples, thus obtaining Table 3, where are shown the results for the first test which was carried out immediately after curing, and the second test after 3 weeks. Fig. 5 highlights the transition from hydrophobic (contact angle >90°) to hydrophilic (contact angle <90°) with a dotted pink line.

From Table 3, it can be seen that the test that shows the highest initial S/N ratio value is sample 8 (−22.1713 dB), and after 3 weeks is sample 9 (−23.5450 dB). These results correlate with the lowest contact angle values, while sample 7 (−36.5526 dB) initially and sample 1 (−36.6744 dB), after 3 weeks, exhibited the lowest S/N ratio values, corresponding to the highest contact angles.

Through Table 4, it is evident that the optimal combination for wettability is 2.5 % surfactant concentration (level 3) cured at 80 °C (level 2), PEO (level 2), both for wettability measured initially and after 3 weeks. As expected, higher surfactant concentrations resulted in increased wettability. The comparison between the three surfactants and the influence of curing temperature is a novel contribution of this study. The Taguchi method indicated that the optimal curing temperature was 80 °C, and among the three surfactants analysed, PEO had the greatest impact on wettability. Since the optimal sample configuration suggested by the Taguchi method was not part of the original set of samples tested according to the orthogonal array, a separate sample consisting of 2.5 % PEO cured at 80 °C was prepared. This sample

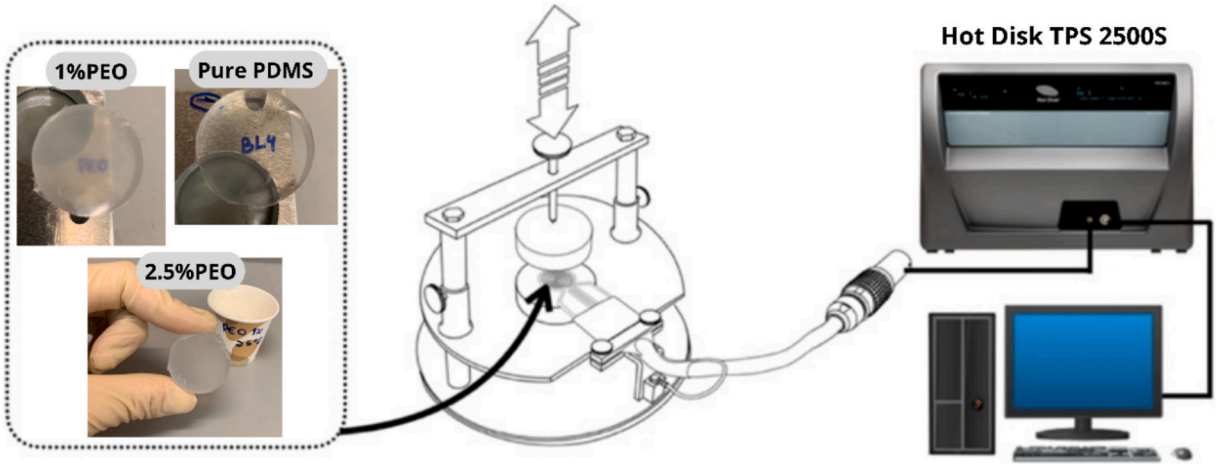

Fig. 4. PDMS sample attached to stainless steel support and Hot Disk TPS 2500S data acquisition system.

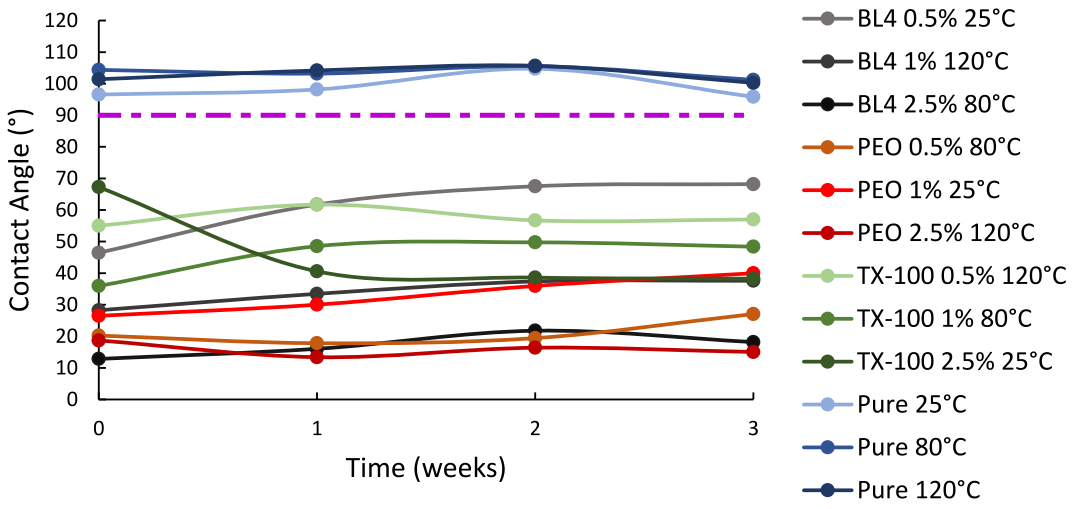

Fig. 5. Contact angle (°) for PDMS samples with different types of surfactants and curing temperatures.

Table 3
S/N ratio value from the initial wettability test (0 h) and after 3 weeks.

Test number	Contact Angle (0 h) (°)	S/N (0 h) (dB)	Contact Angle (3 weeks) (°)	S/N (3 weeks) (dB)
1	46.46 ± 3.06	−33.3416	68.19 ± 4.33	−36.6744
2	20.40 ± 4.36	−26.1926	27.06 ± 3.46	−28.6466
3	55.00 ± 5.03	−34.8073	57.00 ± 5.20	−35.1175
4	25.49 ± 4.00	−28.1274	39.97 ± 3.46	−32.0347
5	35.93 ± 6.00	−31.1091	48.38 ± 4.33	−33.6933
6	28.25 ± 3.21	−29.0204	37.59 ± 5.20	−31.5014
7	67.24 ± 5.51	−36.5526	38.25 ± 3.46	−31.6526
8	12.84 ± 3.06	−22.1713	18.17 ± 4.33	−25.1871
9	13.39 ± 4.16	−22.5356	15.04 ± 3.46	−23.5450

Uncertainties in this table are stated with a 95 % confidence level (k = 2).

exhibited an initial contact angle (0 h) of 12.87°, representing a 62.02 % reduction compared to the average initial contact angle (0 h) of the 9 samples prepared according to the Taguchi array.

In comparison with the contact angle values reported in the literature for various wettability modification techniques and surfactants, the proposed method demonstrates clear advantages. Notably, it promotes a more pronounced and stable increase in hydrophilicity, while also enabling faster recovery from hydrophobic regression than conventional treatments. Several surface modification strategies have been widely employed to enhance the wettability of PDMS, particularly for microfluidic and bioengineering applications. Oxygen plasma treatment, for instance, typically reduces the water contact angle (WCA) to values between 17° and 46°, although these can increase over time to 50°–115° within six hours [14] Other studies have reported WCAs ranging from 40° to 101.17° [44], and around 70° using the SRMJ method [45]. These treatments are often used to improve surface biocompatibility and to modulate cellular interactions. Similarly, UV-Ozone treatment has shown efficacy in lowering WCA to between 10° and 40°, with gradual reversion to 40°–95° over 30 days [46]. A more intensive UV-O exposure reduced the WCA to 7° after 240 min [47]. While these conventional techniques effectively enhance PDMS surface properties, the results obtained in this study indicate that the proposed method, based on the incorporation of surfactants, offers more consistent and durable wettability improvements [9,48], highlighting its potential for advanced applications in microfluidics and related fields.

3.1.2. *ANOVA results*

For the ANOVA, the S/N values calculated using the Taguchi method were used for both wettability and conductivity tests. The analysis was conducted using the S/N values related to the contact angles from the wettability tests to identify the most influential parameters on the surface characteristics of the produced samples. ANOVA was performed with the smallest contact angle recorded during the tests, using the statistical software Minitab 17. The data for this analysis, including the initial contact angle and the angle after 3 weeks, are presented in Table 3. The ANOVA results, initially and after 3 weeks, are detailed in Table 5.

Based on Table 5, the most influential parameter on the contact angle of the samples with surfactant is the type of surfactant, with an influence of almost 55 %. The curing temperature accounts for approximately 28 %, and the surfactant concentration has a 13.6 % influence on the contact angle reduction. After 3 weeks of curing, the most influential factor is the surfactant concentration at 50.57 %, followed by the type of surfactant at 28.88 %, and finally, the curing temperature at 20 %, which aligns with expectations.

Table 5
ANOVA values for the initial wettability test (0 h) and after 3 weeks.

	Initial wettability test	Wettability test after 3 weeks
	% Influence	% Influence
Percentage (%)	13.59	50.57
Temperature (°C)	27.88	20.03
Surfactant Type	54.79	28.88
Error	3.74	0.52
Total	100.00	100.00

Table 4
Average S/N values for parameters and levels of wettability.

Parameters	Initial (0 h)			Final (after 3 weeks)		
	Level 1	Level 2	Level 3	Level 1	Level 2	Level 3
Percentage (%)	−31.4471	−29.4190	**−27.0865**	−33.4795	−32.4098	**−26.7949**
Temperature (°C)	−32.6738	**−26.4910**	−28.7877	−33.4539	**−29.1757**	−30.0546
Surfactant Type	−28.1778	**−25.6185**	−34.1563	−31.1210	**−28.0754**	−33.4878

3.2. Thermal conduction test

3.2.1. Taguchi method results

For thermal conductivity, the goal is to achieve the highest possible value. The S/N ratio for this parameter follows the principle of "higher is better" because it aims to maximize thermal conductivity. Using the values obtained from the thermal conductivity tests and the Taguchi equation, the signal-to-noise ratios for the nine samples were determined, as shown in Table 6. Tests were conducted both immediately after curing and after 3 weeks.

Based on Table 6, the sample with the highest initial S/N ratio value was sample 2 (−12.9025 dB), while sample 1 (−13.9534 dB) had the highest S/N ratio after 3 weeks. These results align with the highest thermal conductivity values and line up with the highest thermal conductivity values. However, the overall differences in thermal conductivity and S/N ratios between samples were minimal, highlighting that PDMS is an insulating material and that surfactants are generally poor thermal conductors. Consequently, the increase in thermal conductivity was low, regardless of surfactant type, concentration, or curing temperature. Table 7 presents the average S/N ratio values for the different parameters concerning the initial thermal conductivity (0 h) and the thermal conductivity after 3 weeks.

As shown in Table 7, the optimal sample, based on the immediate post-curing results, is 0.5 % TX-100 cured at 80 °C. After 3 weeks, the ideal sample is 2.5 % BL4 cured at 25 °C. Although there is a notable difference between the optimal samples immediately after curing and those tested after 3 weeks, the changes in thermal conductivity are relatively minor due to PDMS's inherent insulating properties. The variation among optimal samples for thermal conductivity is much smaller than in the wettability tests, where the selected parameters had a more pronounced impact. It was noted that the variation in the thermal conductivity and S/N values is very low between one sample to another, and this change between the optimal samples for the two tests is not as relevant as when compared to the wettability S/N values, where the parameters that were chosen to be analysed have a major influence on this characteristic.

3.2.2. ANOVA results

Similarly to the wettability test, this analysis was conducted using the S/N values, but this time related to the thermal conductivity test results. ANOVA was performed using the average thermal conductivity values recorded during the tests, with the same software. The data from the initial and 3-week tests are shown in Table 6. The detailed ANOVA results for the initial test and after 3 weeks are provided in Table 8.

As shown in Table 8, the most influential parameter on the thermal conductivity of the samples with surfactant is the residual error, with an influence of approximately 54 %. This error represents the variability that remains in the model after identifying all the main effects [34]. In other words, the selected model fails to explain roughly half of the result variations, suggesting that the chosen parameters and levels may not fully describe the system's behavior. This suggests that the selected parameters may not be ideal for altering the thermal conductivity. Despite the high residual error, the experimental model still showed improvement compared to the initially defined conditions, demonstrating the usefulness of the Taguchi method. After 3 weeks of curing, the most influential factor became the type of surfactant, with an influence of approximately 73 %, followed by surfactant concentration at 10.37 %, and curing temperature at 9.34 %.

Table 6
S/N ratio value from the initial thermal conductivity test (0 h) and after 3 weeks.

Test number	Thermal conductivity (0 h) (W/mK)	S/N (0 h) (dB)	Thermal conductivity (after 3 weeks) (W/mK)	S/N (after 3 weeks) (dB)
1	0.2106 ± 0.0139	−13.5308	0.2006 ± 0.0132	−13.9534
2	0.2264 ± 0.0149	−12.9025	0.1966 ± 0.0130	−14.1283
3	0.1975 ± 0.0130	−14.0887	0.1972 ± 0.0130	−14.1019
4	0.1990 ± 0.0131	−14.0229	0.1952 ± 0.0129	−14.1904
5	0.2116 ± 0.0140	−13.4897	0.1980 ± 0.0131	−14.0667
6	0.2031 ± 0.0134	−13.8458	0.1981 ± 0.0132	−14.0623
7	0.2250 ± 0.0149	−12.9563	0.1993 ± 0.0131	−14.0099
8	0.2000 ± 0.0132	−13.9794	0.1993 ± 0.0132	−14.0099
9	0.1970 ± 0.0130	−14.1107	0.1962 ± 0.0130	−14.1460

Uncertainties in this table are stated with a 95 % confidence level (k = 2).

3.3. Grey relational analysis of the tests

According to the Grey Relational Analysis method, the S/N ratio values from the wettability and thermal conductivity tests, calculated for the PDMS samples with surfactant using the Taguchi method, were used as presented previously.

3.3.1. Grey relational analysis of initial tests (0 h)

The first step in the Grey relational analysis involves normalizing the data. This process aims to eliminate variations in the responses and make them dimensionless by standardizing the results on a scale from 0 to 1. The normalization of the S/N ratio is done using the "larger is better" characteristic since the values used to calculate the Grey relational analysis were the S/N ratios. The "larger is better" and "smaller is better" conditions were already established for each test in the Taguchi method, so the best S/N result will always be the largest. If the means of the values obtained in the tests were used, it would be necessary to apply the condition for "smaller is better" or "larger is better", depending on the test objective [34].

Subsequently, the Grey relational coefficient was calculated. First, $\left|x^0{}_i - x_{ij}\right|$ is determined which represents the difference between the ideal sequence value (the optimal value for the normalization of the S/N, i.e., the ideal S/N ratio result) and the normalized value obtained for each experiment, and each analysed response. Based on these values, the Grey relational coefficients were determined. For this analysis, the distinctive coefficient (ζ) was set to 0.5 for both wettability and thermal conductivity test results, ensuring equal weighting of the optimal conditions for both properties. Finally, the Grey Relational Grade was calculated. This leads to Table 9, which shows the average Grey's relational grade for each level. With this information, it is possible to identify the best combination, and the higher the value of Grey's relational grade, the better the combination.

Table 9 shows that the best combination (optimal sample) of parameters obtained from the Grey analysis is a concentration of 2.5 % (level 3), a curing temperature of 80 °C (level 2) and the surfactant type PEO (level 2). This is the same optimal sample indicated by the Taguchi method, for initial wetting and after 3 weeks. This demonstrates that the selected parameters and levels are more suitable for altering wettability, resulting in a significantly greater improvement in this property than in thermal conductivity. Even with equal weights for both properties (50 % for each), the S/N ratio for wettability had a greater influence.

3.3.1.1. Confirmation of initial Grey's results (0 h). After determining the optimal parameter level and confirming that this configuration had not been tested in all the experiments planned by the Taguchi orthogonal array, additional tests were conducted to validate the results. First, the estimated Grey relational grade for the optimal combination was 2.5% – 80 °C - PEO. Subsequently, three tests were conducted using this optimal combination, referred to as the Optimal Sample Counterproof. From the wettability and thermal conductivity test results, the S/N ratio of each test was calculated.

Applying the same methodology used previously, the Grey relational analysis was performed using the data obtained from the counterproof tests, determining the Grey Relational Grade of the experiment. The average of the initial Grey Relational Grades (0 h), the estimated Grey Relational Grade by calculation, and the Grey Relational Grade from the

Table 7
Average S/N values for parameters and levels of thermal conductivity.

Thermal conductivity						
Parameters	Initial (0 h)			Final (after 3 weeks)		
	Level 1	Level 2	Level 3	Level 1	Level 2	Level 3
Percentage (%)	**−13.5073**	−13.7861	−13.6821	−14.0612	−14.1065	**−14.0552**
Temperature (°C)	−13.5034	**−13.4572**	−14.0150	**−14.0512**	−14.0683	−14.1034
Surfactant Type	−13.7853	−13.6787	**−13.5116**	**−14.0085**	−14.1549	−14.0595

Table 8
ANOVA values for the initial thermal conductivity test (0 h) and after 3 weeks.

	Initial thermal conductivity test	Final thermal conductivity test
	% Influence	% Influence
Percentage (%)	6.73	10.37
Temperature (°C)	32.50	9.35
Surfactant Type	6.46	72.94
Error	54.31	7.34
Total	100.00	100.00

Table 9
Response table for the initial Grey Relational Grade (0 h).

Parameters	Level 1	Level 2	Level 3
Percentage (%)	0.5372	0.4588	**0.6493**
Temperature (°C)	0.5051	**0.6589**	0.4813
Surfactant Type	0.5240	**0.6373**	0.4841

counterproof tests were analysed. Additionally, the percentage improvement between the calculated prognosis and the counterproof results of the optimal combination was compared relative to the average of the Grey relational grades. The average initial Grey Relational Grade (0 h) was 0.5484. The Grey Relational Grade obtained from the counterproof tests was 0.8325, representing a 52 % improvement relative to the initial average. The ideal Grey Relational Grade, calculated theoretically, was 0.8487, showing a 55 % improvement. These results are very close, indicating that the theoretical value aligns with the experimental outcome.

3.3.1.2. ANOVA analysis results. ANOVA was performed on the Grey ratio values obtained in the initial wettability and thermal conductivity tests (0 h). The analysis revealed the percentage influence of each factor on the results.

The surfactant was the most influential factor, accounting for 77.48 % of the variation. Temperature had a significant impact as well, contributing 17.40 %. The percentage factor showed a smaller influence at 3.15 %, while the error accounted for 1.97 % of the total variation.

3.3.2. Grey's relational analysis for tests after 3 weeks

The Grey's ratio analysis of the final tests, after 3 weeks, was performed similarly to the initial tests, using the S/N ratio values obtained. As in the initial tests, a distinctive coefficient (ζ) of 0.5 was used for each test.

Table 10 presents the average Grey's relational grade for each level. The best combination is identified based on the highest Grey Relational Grade, which equally weighs both tests. The optimal combination for both tests is the sample with a 2.5 % surfactant concentration (level 3), at a curing temperature of 80 °C (level 2), and using BL4 surfactant (level 1). This optimal combination was 37 % better than the average of Grey's relational grades.

3.3.2.1. ANOVA analysis results. Similarly to the initial tests, ANOVA

Table 10
Response table for Final Grey Relational Grade for final wettability and thermal conductivity tests after 3 weeks.

Parameters	Level 1	Level 2	Level 3
Percentage (%)	0.5176	0.4411	0.6638
Temperature (°C)	0.5379	0.5580	0.5266
Surfactant Type	0.6306	0.5195	0.4724

was conducted on the Grey relational grade values obtained from the tests carried out after 3 weeks of curing. The results show that the factor that most influenced the combined wettability and thermal conductivity after 3 weeks of curing was the surfactant concentration in the PDMS surfactant mixture, with approximately 57 % influence, followed by surfactant type at 29.2 %, and the curing temperature at only 1.1 % and a final error of 13 %. The change in parameter influence compared to the initial tests may be due to the selection of parameters unsuitable for thermal conductivity, resulting in unexpected variations.

4. Validation of the optimal wetting sample using the Taguchi method

Based on the optimal sample identified using the Taguchi method, an application was then performed with the parameters of this optimal sample to validate the result. The best sample from the wettability test was chosen, as there was a significant improvement in this property, while the improvement in thermal conductivity was negligible. For this reason, the validation focused on wettability, rather than on thermal conductivity or Grey's relational analysis, which considered 50 % wettability and 50 % thermal conductivity, thus heavily influencing the thermal conductivity portion.

It was decided to apply the sample to microchannels, comparing pure PDMS with the optimal sample for wettability from the Taguchi method, a sample with 2.5 % PEO cured at 80 °C, to analyse the improvement. Increased hydrophilicity in PDMS is essential for microchannel applications because it enhances fluid flow through capillarity, allowing liquids to move through microchannels without pumps or forced movement devices. Additionally, improved hydrophilicity reduces protein and cell adsorption on channel walls and increases compatibility with various aqueous reagents, optimizing the performance of microfluidic devices and simplifying their design and operation [23–25].

The microchannels were fabricated using the same method as for the samples, with the difference being that instead of pouring the mixture and the pure PDMS into the circular mould, it was poured into the microchannel mould (Fig. 2).

4.1. Capillary flow studies

Capillary flow tests were conducted to evaluate surface wettability by observing the self-movement of fluid through the devices. A small volume of water ($v = 100$ μL) was pipetted at the inlet, and its flow was monitored by measuring the time, in seconds, required for the liquid to reach the outlet of the device (Fig. 6). The PDMS microchannels were fabricated using both control (pure) and bulk-modified PDMS. The results from the control samples showed that the hydrophobic nature of

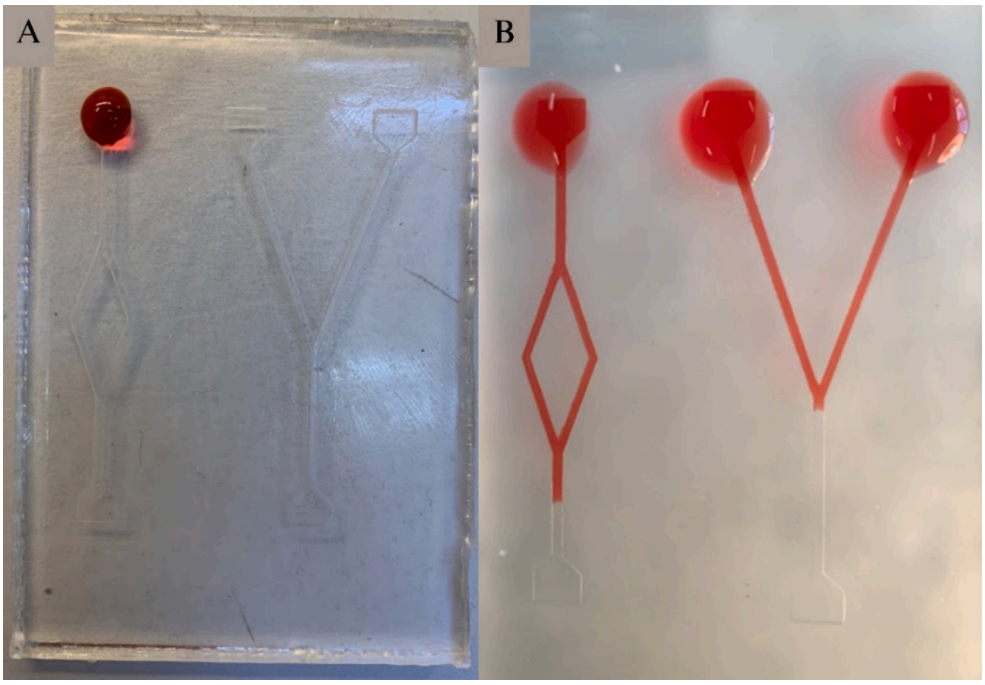

Fig. 6. Microchannel samples with a 10:1 ratio in (A) pure PDMS (control), and (B) PDMS mixed with 2.5 % PEO surfactant cured at 80 °C.

PDMS hinders the flow of a liquid, in this case, distilled water, through the channels (Fig. 6A). Subsequent tests on the devices with added surfactant (Fig. 6B) clearly showed the rapid and immediate movement of the liquid, with the results presented in Table 11. Three tests were carried out on each microchannel, and the averages were calculated. The tests involved pure PDMS (control) and 2.5 % PEO cured at 80 °C, the optimal sample for wettability, immediately after curing (0 h) and after 24 h of curing. The fluid flow time is recorded in seconds.

The results demonstrated that the samples modified with 2.5 % PEO cured at 80 °C showed a very considerable improvement compared to pure PDMS (control). However, compared to studies that were performed in similar microchannels, but which were closed, a research work developed by Gonçalves et al. [28]. The improvement was lower, showing that the wettability for the same concentration and type of surfactant in open microchannels has a lower flow compared to closed micro-channels. According to the data in Table 11, the mixture became more hydrophilic after 12 h of curing, likely due to the stabilization of the PDMS/surfactant mixture, which may benefit from an extended curing time beyond the manufacturer's recommendation for the selected curing temperatures. Furthermore, after 24 h of curing, there was minimal hydrophobic recovery according to the capillary flow data presented, suggesting that this modification could be suitable for modifying the PDMS surface for in vitro studies.

It is also worth noting that the flow times in the microchannels presented in this study are longer than those observed in closed microchannel studies. This can be explained by the fact that closed microchannels have a hydrophilic material covering their entire surface, which facilitates fluid flow. In contrast, open microchannels only have hydrophilic properties on the base and sides.

Table 11
Results of capillary experiments on microchannels with test 1 (0 h), test 2 (12h) and test 3 (24 h) after curing. The fluid flow time is measured in seconds.

Microchannels	Time (seconds)				
	Control PDMS		PEO 2.5 % 80 °C		
	Test 1	Test 3	Test 1	Test 2	Test 3
A	–*	–*	227.0	154.7	171.0
B	–*	–*	160.5	122.3	119.3
C	–*	–*	151.0	128.3	152.7
D	–*	–*	190.3	142.3	168.3

* Due to the hydrophobic nature of PDMS, no fluid movement was observed in the control samples.

5. Conclusions

This study aimed to optimize the wettability and thermal conductivity parameters of samples containing three types of surfactants: PEO, BL4 and TX-100. Three different concentration levels of these surfactants were analysed, along with variations in curing temperature, using contact angle measurements to assess wettability and standardized criteria for thermal conductivity. The Taguchi method, Grey Relational Analysis, and ANOVA were used to evaluate the influence of each parameter on the results.

The results showed that the optimal combination for wettability is 2.5 % PEO cured at 80 °C, both initially and after three weeks. Analysis of the S/N value averages showed that higher surfactant concentrations increase wettability. Among the selected parameters, the surfactant type had the most significant influence, followed by the curing temperature and, finally, the surfactant concentration. Surfactant type affected the initial contact angle by nearly 55 %, while curing temperature and surfactant concentration accounted for 28 % and 13.6 %, respectively. After three weeks, surfactant concentration became the most influential factor with 50.57 %, followed by surfactant type with 28.88 % and curing temperature with 20 %.

Concerning thermal conductivity, the best initial combination was 0.5 % TX-100 cured at 80 °C, and after three weeks, it was 2.5 % BL4 cured at 25 °C. However, the variation in thermal conductivity was less

significant compared to wettability, as the chosen parameters were not as effective for optimizing this property. Grey's analysis of the initial tests (0 h) confirmed that the optimal combination, considering both tests, is 2.5 % PEO cured at 80 °C. This combination showed a 52 % improvement over the other samples produced using the orthogonal Taguchi array. After three weeks, Grey's analysis pointed to 2.5 % BL4 cured at 80 °C, with ANOVA showing that surfactant concentration was the most influential factor (approximately 57 %), followed by surfactant type (29.2 %) and curing temperature (1.1 %). Based on the test results, the validation focused on wettability because of the significant improvement in contact angle reduction. In contrast, thermal conductivity showed minimal improvement. As a result, the sample with 2.5 % PEO cured at 80 °C was used to manufacture microchannels. Increased hydrophilicity in PDMS is essential for microchannel applications, as it improves fluid flow through capillarity, among other applications, optimizing the performance of microfluidic devices. The results showed that samples modified with 2.5 % PEO and cured at 80 °C performed significantly better than pure PDMS but were still less effective compared to closed microchannels. The mixture became more hydrophilic after 12 h of curing, with stabilization of the PDMS/surfactant mixture. After 24 h, there was low hydrophobic recovery, suggesting that this modification may be suitable for in vitro studies. Additionally, flow times in open microchannels were longer than in closed microchannels, due to differences in surface hydrophilic. The effectiveness of the Taguchi method in optimizing wettability parameters was validated, showing the importance of considering both wettability and thermal conductivity in future studies and practical applications.

CRediT authorship contribution statement

Lucas B. Neves: Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Data curation. **Inês S. Afonso:** Writing – review & editing, Visualization, Investigation, Formal analysis. **Luiz G. Barbosa:** Writing – review & editing, Supervision, Investigation, Formal analysis. **Glauco Nobrega:** Writing – review & editing, Visualization, Validation, Investigation, Formal analysis. **Rui A. Lima:** Writing – review & editing, Supervision, Resources, Methodology, Funding acquisition, Conceptualization. **João E. Ribeiro:** Writing – review & editing, Validation, Supervision, Resources, Project administration, Methodology, Funding acquisition, Formal analysis, Conceptualization.

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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.

Data availability

No data was used for the research described in the article.

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