

RELATIVE WETTABILITY MEASUREMENT OF POROUS DIFFUSER AND ITS IMPACT ON THE GENERATED BUBBLE SIZE

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Controlling the bubble size is a major concern in enhancing transport performance in gas-liquid systems. The role of wettability of diffuser surface on bubble size is the subject of the current work. The study inspects the contact angle of a set of liquids on HP ceramic diffusers using the Washburn method. The results demonstrate that organic liquids like toluene, methanol–water (1:1 v/v), ethanol–water (1:1 v/v) and decane have small contact angles of 12.9°, 37.5°, 24.4° and 22.5° respectively. Water has a lower wettability than the organic compounds where the contact angle was about 67.4°. The effect of wettability of the bubble size is investigated by measuring the size of air bubble produced using the same diffuser material. The results of bubble size measurement demonstrates that with liquids of small contact angle, i.e. good wetting properties, small bubble sizes are produced in comparison with liquids with a higher contact angle. The study demonstrates the viability of Washburn method in characterization of wettability of porous diffuser, which was verified by measuring the bubble size produced. A high reduction in bubble size can be obtained by a carefully chosen diffuser material that provides better wettability.

Keywords: bubble size, contact angle, porous diffuser, Washburn method, wettability

1. INTRODUCTION

In bubble columns, bubble size plays a critical role in the separation process efficiency. The rates of mass and heat transfer are proportional to the interfacial area of the gas bubbles. The specific gas–liquid interfacial area is inversely proportional to the bubble diameter (Kantarci et al., 2005), hence, small bubble size enhances the transfer process by providing a high transfer surface area to volume ratio (Worden and Bredwell, 1998; Zimmerman et al., 2011a). Additionally, small bubble sizes have extraordinary features like slow rising time and intensive internal mixing. This prompts researchers to explore important industrial applications involving micro/nano scale bubbles (Al-yaqoobi et al., 2016; Hanotu et al., 2014, 2013; Nashmi et al., 2020; Zimmerman et al., 2011b).

One may think that reducing the diffuser pore size is enough to produce small bubbles, but it is found conventionally that the bubble radius is nearly three orders of magnitude larger than the radius of orifice

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producing it (Liow and Gray, 1988; Zimmerman et al., 2011a). However, there are other factors that affect the bubble formation mechanism. In the steady flow regime, the bubble continues to grow on the pore surface until the buoyant force (which is proportional to the bubble volume) overcomes the surface tension force. Before the bubble breaks off from the diffuser surface, the difference between upward forces and downward forces is a key factor for the bubble detachment from the diffuser. Consequently, the wetting property of the diffuser material has an essential role in controlling the bubble size formation (Byakova et al., 2003; Zimmerman et al., 2011a).

Wettability is the tendency of a liquid to spread on or adhere to a solid surface in the presence of other immiscible fluids. The contact angle, θ , is the measure of wettability of a solid by a liquid (dos Reis et al., 2009). When the contact angle is less than 90° , good surface wettability is exerted; conversely, a poor wettability correlates with a contact angle greater than 90° .

Different methods have been used to determine the wettability by utilizing direct and indirect measurement of the contact angle. Sessile drop and pendant drop are direct methods, which require a flat and smooth surface (Shang et al., 2008). If the surface is porous, the liquid drop penetrates the solid surface. Consequently, alteration of the contact angle with time will be observed. Liquid could imbibe into the pore, which influences the observed contact angle. The Washburn or capillary-rise method is an indirect method which can be used to characterize the contact angle of porous solids or powder (Alghunaim et al., 2016).

The Washburn method is a straightforward method to measure the wettability. The Washburn equation describes the height of the liquid, h , rising in a straight linear capillary of radius, r , as a function of time, t :

$$\frac{h^2}{t} = \frac{r\gamma_l \cos \theta}{2\mu} \quad (1)$$

where μ is the liquid viscosity, γ_l is the surface tension, and θ is the contact angle. When Washburn's theory is applied for porous media, the sample is assumed to be a bundle of cylindrical capillaries and the mean equivalent pore radius is equal to the capillary radius.

Several conditions should be satisfied to determine the contact angle when Washburn method is used for powders or porous materials. These conditions can be summarised as follows: (i) steady-state laminar flow of the imbibition motion is assumed, (ii) zero velocity of the liquid at solid/liquid interface (no slip), (iii) no applied pressure, and (iv) negligible gravitational differences (Kirdponpattara et al., 2013; Siebold et al., 1997).

The principle of modified Washburn's method is based on the measurement of the liquid weight during the penetrating time. The liquid mass, m , is related to the height by the relation:

$$m = \varepsilon \rho A h \quad (2)$$

Where ε is the porosity of the surface, ρ is the density of liquid, and A is the cross-sectional area of the packed tube.

From the previous relationships, the following equation can be written as:

$$\frac{m^2}{t} = \frac{C\rho^2\gamma \cos \theta}{\mu} \quad (3)$$

where C is the geometric factor (or material constant), which is represented by:

$$C = \frac{rA^2\varepsilon^2}{2} \quad (4)$$

The geometric factor is constant as long as the sample geometry and porosity remain constant.

According to the modified Washburn theory, when a porous sample is brought into contact with a liquid, the rise of the liquid in the pores is governed by Equation (3).

The results are obtained by plotting the square of the adsorbed liquid mass (m^2) as a function of time, t , which gives a slope of $(C\rho^2\gamma \cos \theta/\mu)$. If the liquid properties are known (i.e. its viscosity, density and surface tension), the only two unknown values are contact angle, θ , and the geometric factor of the sample, C , (Neirinck et al., 2010; Nowak et al., 2013).

For a certain porous material, the geometric factor, C , could be evaluated experimentally by using a liquid that has a low surface tension such as n-hexane (surface tension of $18 \text{ mN}\cdot\text{m}^{-1}$). Such liquids have a high wetting ability (complete wetting of the material) and hence the contact angle could be assumed to be zero.

Consequently, the Washburn wettability method involves two steps. First, finding the geometric factor, C , with a completely wetting liquid. Second, determining the contact angle for various liquids by the assumption of constant geometric factor (Galet et al., 2010; Rulison, 1996).

The aim of this study is to investigate the wettability of a set of liquids on a HP ceramic diffuser by using Washburn method. The liquids used in this work were water, hexane, toluene, 50%(v/v) ethanol aqueous solution, 50%(v/v) methanol aqueous solution, and decane. Thereafter, the effect of the wettability of the diffuser material by the tested liquid on the generated bubble size was investigated. For the aforementioned purposes, two laboratory systems were configured to conduct the experimental work for the wettability tests and the bubble size measurements.

This paper is organised as follows: the materials, experimental methodology and equipment used in the work is presented in Section 2. The results of the experiments and the discussion is explained in Section 3. The last section is dedicated to conclusions.

2. MATERIALS AND METHODS

Ceramics are popular materials in manufacturing diffusers. In this work, HP Technical Ceramics, UK, sintered microporous alumina-silica ceramics was chosen to investigate the wettability and the generation of bubbles. The dimensions of the sample used in this work were $5.6 \text{ cm} \times 2.5 \text{ cm} \times 1 \text{ cm}$. The chemical composition of the diffuser material was as follows: SiO_2 (81%), B_2O_3 (12%), $\text{Na}_2\text{O} + \text{K}_2\text{O}$ (4%), Al_2O_3 (2%).

The liquids used in this work were: distilled water, hexane, toluene, 50 % (v/v) methanol–water solution, 50% (v/v) ethanol–water solution and decane, which were purchased from Sigma–Aldrich. The properties of the tested liquids are listed in Table 1.

The experimental procedure for the wettability measurements consists of two parts: the first part includes cleaning, drying and cooling of the samples. The second part was measuring the liquid imbibition rate to estimate the contact angle between tested liquid and the diffuser samples. After that, the bubble size measurement is conducted by imaging the bubble produced through the HP diffuser.

The diffuser sample cleaning was conducted using an ultrasonic bath (Jaken PS-08A) and the cleaning process lasted for 30-60 minutes according to the tested liquid. A 20% ethanol–water solution was used to wash the samples.

A furnace at $80 \text{ }^\circ\text{C}$ was used to dry the diffuser sample for 1 hour. After that, the sample was cooled by air and kept at room temperature for 30 minutes. It is important to mention that the initial weight of

Table 1. Properties of liquids used in the work

Tested liquid	Density [kg/m ³]	Viscosity [mPa·s]	Surface tension [mJ/m ²]
Water	998	1.002	72.3
Hexane	664	0.326	18.7
Toluene	862	0.55	27.8
50% Ethanol	921	2.7	30.6
50% Methanol	937.2	1.542	33
Decane	730	0.92	23.9

sample was checked before each run to be sure that the cleaning and drying process were accomplished successfully.

Wettability experiments were performed using the apparatus shown in Fig. 1. After the sample is cleaned and dried as described before; the sample is fixed on sample holder, which allows the sample to touch the liquid surface steadily. The sample is hinged by a screw, which allows the sample to be moved up and down. The sample holder is fixed on a bridge, which is clamped on a stand. The liquid container is placed on a sensitive balance (Sartorius BP210S) to measure the liquid weight every second. The balance is connected to a computer by RS232 to USB converter. With this apparatus, the samples come into contact with surface of tested liquid and the weight loss of the liquid is recorded as a function of time. The data recording proceeds until the sample detaches from the liquid surface, or when the curve trend begins to level off. After each measurement of imbibition rate, the diffuser sample is prepared for the next round of measurement as described before.

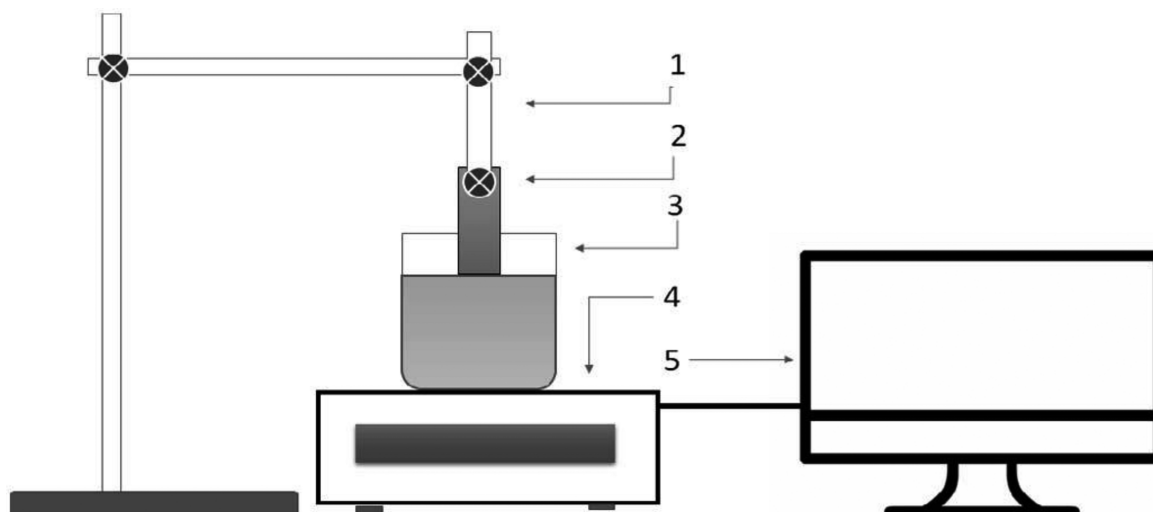


Fig. 1. Schematic diagram of wettability test apparatus: 1 – sample holder, 2 – porous material sample, 3 – tested liquid container, 4 – digital balance, 5 – PC

The imaging of bubble generation by HP diffuser is conducted by using the apparatus shown in Fig. 2. A glass box of 14 cm × 10 cm × 18 cm dimensions is used as the bubble column. The box is located over a flat base where the HP ceramics diffuser is fixed. The air is pumped to the tank from the bottom and introduced to the liquid through the diffuser. A flowmeter is connected to the gas outlet at the top of the tank. A high-speed camera (Photron SA-3) is used for bubble imaging. Several images were captured

at a rate of 2000 frames/sec. The light is provided by two halogen lamps (Model no: HM-682C; 150 W Argos, UK), which are fixed on either side of the camera.

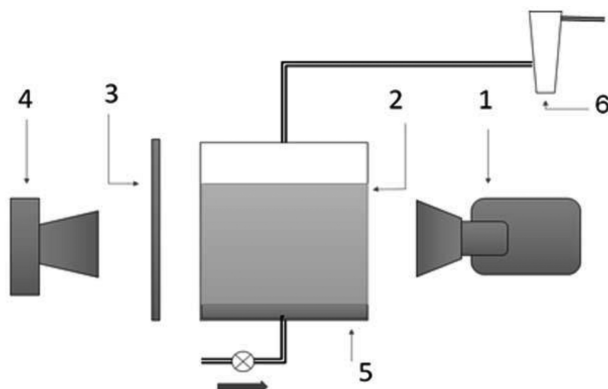


Fig. 2. Schematic diagram of bubble imaging apparatus: 1 – camera, 2 – glass tank, 3 – light diffuser, 4 – light source, 5 – diffuser, 6 – flowmeter

3. RESULTS AND DISCUSSION

Weight versus time measurements were chosen instead of imaging of liquid progression through the sample material, which was deemed too imprecise. Significant uncertainty in liquid height progression is indicated in experiments conducted with powder packing sample (Kilau and Pahlman, 1987; Varadaraj et al., 1994). The curves presented in Fig. 3 show the behaviour of the liquid weight versus time for the different tested liquids.

Based on the behaviour of the liquid imbibition rate that is shown in Fig. 3, two stages of liquid mass penetration are distinguished. First, a sharp increase in liquid mass imbibes through the sample, thereafter; it gradually reached more or less a constant value until the sample detached from the liquid. From Fig. 3 it can be indicated that the relationship between the square of liquid mass and penetration time is linear during the initial rise, which satisfies Washburn equation, and it was observed in all tested liquids. Clearly, the curves all have a sigmoidal shape as they approach the plateau where imbibition level equilibrates. The Washburn theory only applies in the linear initial phase of the curves.

The behaviour of liquid imbibition rate could be attributed to the domination of inertial forces in early penetration time, after that a viscous regime is identified where Washburn's equation can be applied (Quéré, 1997). In the second stage, the trend of the wettability curves started to flatten because the pores of the sample material are completely filled with the tested liquid. However, for some liquids, the material samples detach from the liquid interface before the plateau stage is reached, as observed with toluene in Fig. 3c.

In order to determine the slope from the obtained curves the linear part of the curves were fitted using the method of least squares (Galet et al., 2010, Siebold et al., 1997). In (Fig. 3a), the square weight of hexane versus time gave a slope of 0.31. This value resulted in a geometry factor, C , of 0.01257×10^{-12} , which was determined according to Equation (4). Subsequently, the contact angle can be estimated for all tested liquids as the geometry factor is known. Table 2 demonstrated the slopes of the linear part of the curves and the calculated contact angles for the tested liquids that were presented in Fig. 3.

The linear region of the curves yields a correlation factor of at least 0.99 for all tested liquids for linear regression. The reproducibility of experiments is demonstrated by repeating the experiment three times. It was found that the values of contact angle obtained varied within 2–3% for all liquids. The porous sample

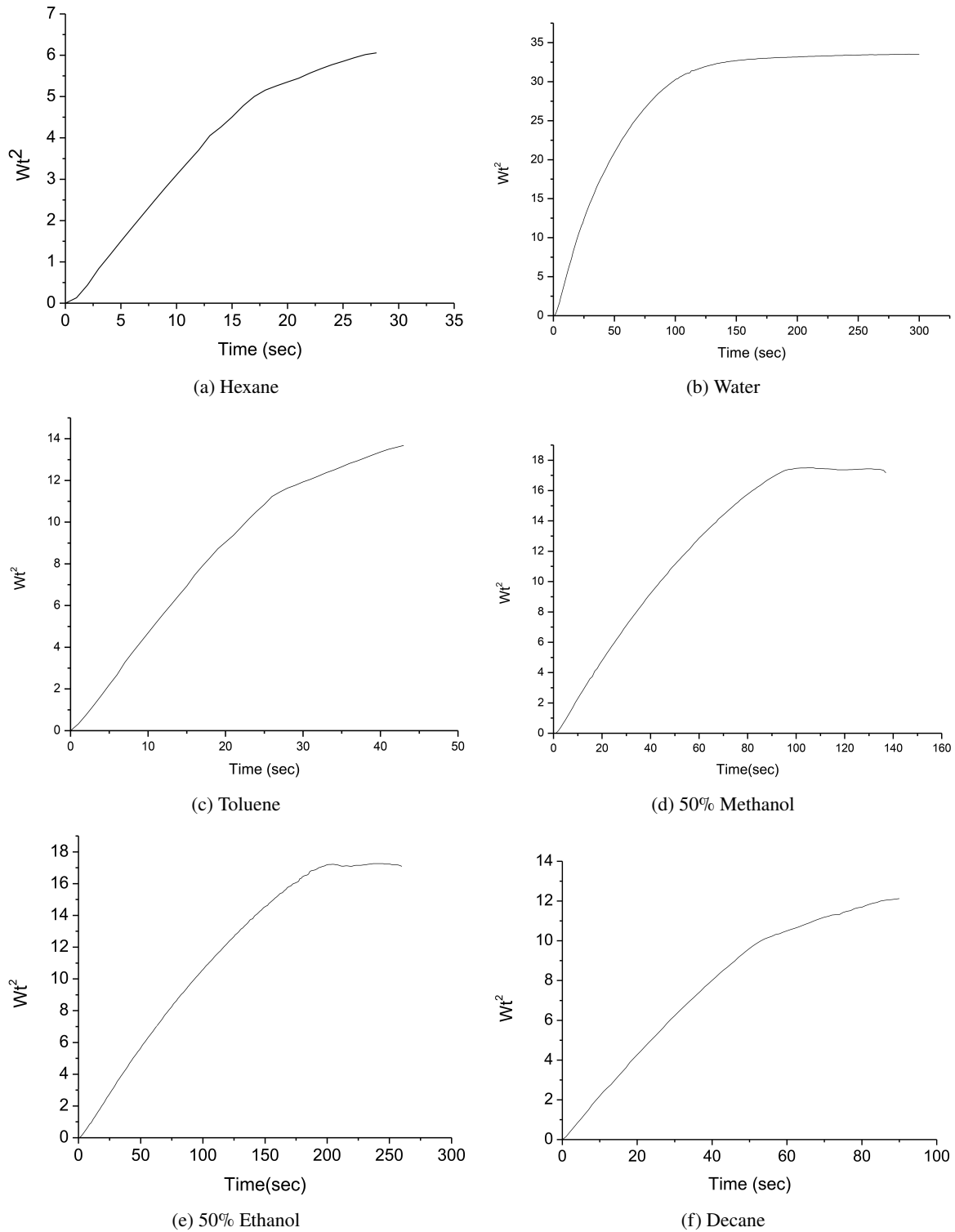


Fig. 3. Weight of liquid penetrating of set of liquid with penetration time. Where the mass of liquid imbibition wt. in [g]

used in the current study is rigid with constant dimension, which limits the error due to variation in the porosity and pore size distribution (Alghunaim et al., 2016; Klein et al., 2012).

The results in Table 2 show that the penetration rate was not in direct correlation to wettability of solid, which could be due to the effect of liquid properties (i.e. viscosity, surface tension, and density) (Dang-Vu and Hupka, 2005).

Table 2. Contact angles and geometry factor (C) of HP diffuser with different tested liquids

Testing liquids	Slope [g ² /sec]	Contact (C) ×10 ⁻¹² [m ⁵]	Contact angle [deg.]
Hexane	0.31	0.01257	×
Water	0.34	0.01257	67.4
Toluene	0.46	0.01257	12.99
50% Methanol solution	0.187	0.01257	37.5
50% Ethanol solution	0.11	0.01257	24.4
Decane	0.161	0.01257	22.5

It can be observed that the lowest contact angle of 13° was achieved with toluene that has a high penetration rate (slope of 0.46), while the highest contact angle of 67.4° was observed with water which has a lower penetration rate than toluene. Adding alcohols (methanol and ethanol) to water resulted in a higher wettability for the mixture. Therefore, the contact angle of 50% ethanol solution reached 24.4° and of 50% methanol solution reached 37.5°. Both values are much lower than that of water. The contact angle decreases with the number of carbon atoms of the tested liquid. This behaviour was also detected in previous studies (Dang-Vu and Hupka, 2005; Kalogianni et al., 2004; Siebold et al., 2000).

In order to inspect the effect of wetting properties on the bubble size, experiments were conducted to measure the bubble size produced in selected liquids. Water, 50% ethanol–water and 50% methanol–water solutions were chosen to investigate the effect of wettability on the bubble size. The three tested liquid have different contact angles (water 67.4°, ethanol–water 24.4° and methanol–water 37.5°). Air bubbles are introduced into the solutions by using the same diffuser material that is used in the wettability test (HP ceramic). The size of the bubbles produced with the chosen liquid is likely to demonstrate the effect of wettability on produced bubble size.

Figure 4 shows the stream of air bubbles in the tested liquids. The airflow rate is constant at approximately 0.05 L/min, the experimental conditions are kept constant except the tested liquid. The bubble size distributions for water, methanol–water and ethanol–water are presented in Fig. 4. It can be observed from the bubble size analysis that 51% of the produced bubbles in water was in the range of 700–900 µm, with Sauter mean bubble size of 793 µm. The bubbles produced in methanol–water mixture had a higher peak of 22% for the bubble sizes of 300–350 µm. It also can be noticed that 44% of the bubbles produced had average sizes of 350–700 µm, while 34% of the bubbles had average bubble sizes of less than 300 µm. For such a system the Sauter mean bubble size was around 380 µm. For the ethanol–water mixture, 21.5% of the bubbles had the average sizes of 250–300 µm. Most of the bubbles (53.1%) had average bubble sizes of less than 300 µm. On the other hands, only 29.9% of the bubbles had average sizes of 350–700 µm. The Sauter mean bubble size was around 372 µm with the ethanol–water mixture. Obviously, a large proportion of the bubbles produced (53.1%) with ethanol–water system were less than 300 µm, which is much smaller than those produced with methanol–water mixture. A high population of bubbles produced in methanol–water were shifted toward average bubble ring of 350–700 with total bubble frequency of 44%. However, the bubbles produced in water were much bigger than those produced with both ethanol–water and methanol–water mixtures.

The contact angle of 50% ethanol on HP ceramic diffuser was 24.4°, which is smaller than that obtained with methanol–water (37.5°) and water (67.4°) as listed in Table 2. In other words the wettability of 50% for ethanol solution is better than that of methanol–water and water. Eventually, the bubble size distributions of the ethanol–water and methanol–water are not remarkably diverse from each other, but the bubble size

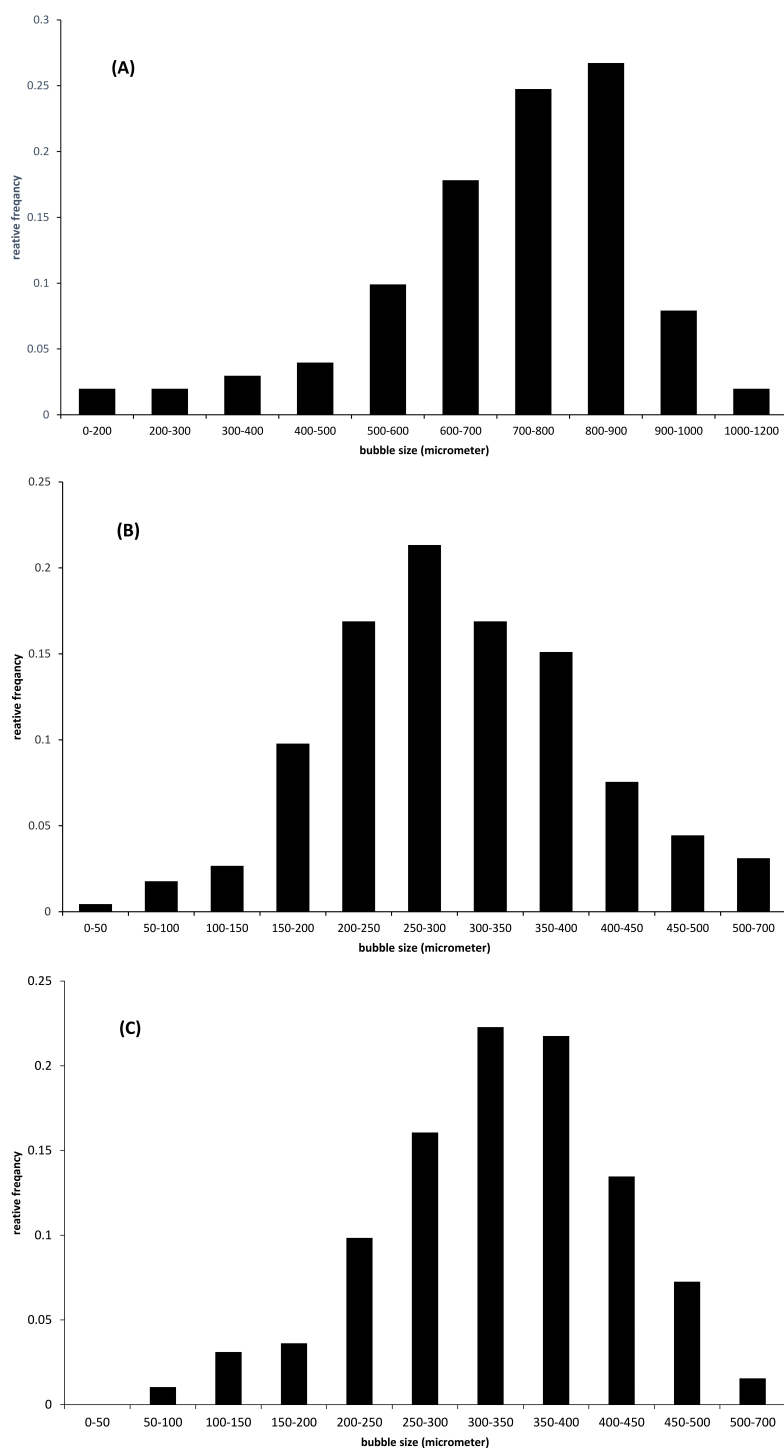


Fig. 4. Bubble size distribution in A – water and B – ethanol–water, C – methanol–water using HP ceramic diffuser

produced with both solutions is obviously smaller than that of water. The bubble size distribution of the studied systems in Fig. 4 shows a good agreement with their values of contact angles as in Table 2.

The results demonstrated that when the contact angle is small (good wettability) the liquid displaces the air from the diffuser surface. That minimizes the downward force acting on the bubble so a small volume of blowing air (upward force) is sufficient to detach the bubble with minimum volume. On the other hand, when the contact angle is large (weak wettability) the air is spread on a larger area on the diffuser by crowding out the liquid from the surface. Consequently, the bubble grows until the volume is large enough to produce an adequate upward force for bubble detachment.

The current results agree with the prior investigations which explored the effect of wettability on the formation of bubble size. [Mirsandi et al.\(2020\)](#) studied the effect of wettability of ethanol–water solutions (0–10%) on the bubble size produced by submerged orifices. Their results show that the contact angle increased with increasing the surface tension of the solution. Also, they demonstrated that the bubble volume increased with increasing the surface tension (reducing ethanol concentration). The contact angle was measured with Sessile drop method. The influence of wetting conditions on bubble formation on an orifice submerged in a water–soap solution of contact angle ranging between 15° and 110° was also investigated by [Byakova et al. \(2003\)](#). The study concluded that the bubble volume increased as the wettability weakened. On the other hand, [Chen et al. \(2002\)](#) explored the impact of solid sparger wettability on bubble size formation. Despite the contact angles being detected properly by the Washburn method, the results failed to show a significant effect of sparger wettability on the size of bubbles produced. [Wesley et al. \(2016\)](#) inspected the impact of wettability on the bubble formation from porous diffuser. The results demonstrated that bubble size was not contingent on the contact angle up to approximately 90°. The contradiction between the results may be due to the diversity of the methods adopted to measure the wettability, the characteristics of the diffuser (pores size, pores size distribution and the composition of the diffuser material) and the techniques used to demonstrate the effect of wettability on bubble size.

4. CONCLUSIONS

In the current study, the wettability of HP ceramic diffuser and the effect of wettability of bubble size were investigated. The Washburn method was used to estimate the contact angle of a set of liquids on the HP diffuser. The results showed that the contact angles of decane, 50% ethanol, 50% methanol were smaller than that of water. Toluene had the smallest contact angle of all tested liquids, which signified that the organic compounds had smaller contact angles than that of water. Consequently, air bubbles produced by the HP diffuser in 50% ethanol solution were smaller than those produced in water. The current work elucidates the efficiency of Washburn method in determining the contact angles of different liquids on porous surface. In addition, the results demonstrate the significance of wettability of liquids on the controlling of bubble size.

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SYMBOLS

A	cross-sectional area of the packed tube, m ²
C	geometric factor, m ⁵
h	Liquid height, m
t	time, sec
r	radius of capillary, m
wt	liquid mass penetration, kg

Greek symbols

μ	liquid viscosity, Pa·s
ρ	density of liquid, kg/m ³

- γ_l surface tension, J/m²
 Θ contact angle, deg.
 ε porosity of the surface, –

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