

# EFFECTS OF MULTIPLE PASSES THROUGH A DYNAMIC MEMBRANE ON THE PROPERTIES OF W/O EMULSIONS

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Conventional membranes used in the process of premix membrane emulsification are prone to fouling, especially when biopolymers are employed as surfactants. An alternative to conventional membranes are dynamic membranes consisting of an unconsolidated porous medium. Dynamic membranes have the advantage of enabling easy cleaning of the inside of the pores. Experimental research carried out to date has focused on the application of hydrophilic dynamic membranes composed of glass microbeads for producing o/w emulsions. The aims of this study were to determine the efficiency of droplet size reduction in a w/o emulsion when passed through a dynamic hydrophobic membrane consisting of a bed of irregular polymer particles, and to assess the effect of multiple membrane passes on the properties of the w/o emulsion. The dynamic membranes evaluated in the tests were found to reduce the diameters of premix droplets when an appropriate pressure level was reached. Higher bed porosity was associated with greater fluxes achieved across the packed bed, but the resulting emulsions were less homogeneous. Multiple passes of the emulsion through the dynamic polypropylene membrane led to a further reduction in droplet size, but it was accompanied by a decline in emulsion homogeneity.

**Keywords:** DMTS system, dynamic membrane, water-in-oil, premix membrane emulsification, membrane homogenization

## 1. INTRODUCTION

Premix membrane emulsification was proposed by Suzuki et al. (1996) in 1996. The process of producing emulsions by this method is divided into two stages. The first stage involves preparing a coarse emulsion (so-called premix) by mixing the ingredients. The premix has a relatively large droplet diameter and a broad distribution of droplet diameters. The emulsion thus prepared is passed through a membrane, leading to a reduction in droplet size. The resulting fine emulsion is characterised by a narrow droplet size distribution, and its preparation requires little energy compared to the methods used conventionally for obtaining emulsions (van der Zwan et al., 2008). Premix membrane emulsification is not only a good alternative to conventional emulsion preparation methods, but also to other membrane-based techniques (Vladislavjević and Williams, 2005) such as direct membrane emulsification and microchannel devices.

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The primary advantage of the method is the possibility to obtain higher transmembrane fluxes. A drawback is the occurrence of depth fouling, especially when biopolymers are used as an emulsifier (Nazir and Vladisavljević, 2021). Membrane methods for obtaining emulsions are suitable in a range of applications, for example in the production of multiple emulsions, food emulsions, drug delivery systems or microcapsules (Vladisavljević and Williams, 2005).

To eliminate membrane fouling, van der Zwan et al. (2008) proposed replacing the conventional membrane with unconsolidated porous media supported by a sieve, obtaining a Dynamic Membranes of Tunable Pore Size (DMTS) system. An advantage of this option is easy cleaning of the inside of the pores. In the initial experimental studies, a packed bed of glass beads was used (van der Zwan et al., 2008). It was characterized by a similar morphology to some of the conventional membranes used for membrane emulsification, while the pore size and tortuosity were close to those found in microporous membranes. Dynamic membranes have proven to be a robust alternative to conventional membranes. Experimental studies conducted to date have been concerned with the application of hydrophilic dynamic membranes consisting of glass beads (Eisinaite et al., 2016; Kaade et al., 2020; Ladjal Ettoumi et al., 2017; Laouini et al., 2014; Nazir et al., 2013; 2014; Sahin et al., 2014; Sawalha et al., 2016; van der Zwan et al., 2008; Wang et al., 2021a; 2021b; 2020). No reports have been found on the preparation of w/o emulsions, the use of hydrophobic membranes, and the application of non-spherical particles.

The objective of this study was to determine the efficiency of droplet size reduction in a w/o emulsion while being passed through a dynamic membrane. A hydrophobic bed of irregular particles of polypropylene (PP), poly(vinyl chloride) (PVC) and polyethylene (PE) was used as a porous bed. In addition, glass beads (GB) studied by earlier researchers were applied for comparison purposes.

## 2. METHODS AND MATERIALS

### 2.1. Measurement of the properties of emulsions and packed beds

Droplet and particle size measurements were performed by taking photographs with a Nikon Eclipse 50i microscope fitted with an OptaTech camera. To avoid the collapse of w/o emulsion droplets, a phenomenon occurring during microscope work, as reported by Sotoyama (1999) hydrophobic microscope slides were used. The microscopic images were analyzed in the Mathworks Matlab R2017b software. For objects with a circular cross-section, the function “*imfindcircles*” was used to obtain their diameters. For irregular objects, the function “*regionprops*” was used to measure the projection area  $S$  and perimeter  $O$ . In the next step, the circle equivalent diameter (Bagheri et al., 2015):

$$d_z = \sqrt{\frac{4 \cdot S}{\pi}} \quad (1)$$

and circularity (Cox, 1927):

$$C = \frac{4 \cdot \pi \cdot S}{O^2} \quad (2)$$

were determined. Based on the known diameters (and equivalent diameters), it was possible to calculate the mean diameters,  $d_{pq}$ , from the general equation:

$$d_{pq} = \sqrt[p-q]{\frac{\sum_{i=1}^m d_i^p \cdot n_i}{\sum_{i=1}^m d_i^q \cdot n_i}} \quad (3)$$

The Sauter mean diameter,  $d_{32}$ , and de Brouckere mean diameter,  $d_{43}$ , were determined. The span value was also calculated (Sahin et al., 2014):

$$\delta = \frac{d_{90} - d_{10}}{d_{50}} \quad (4)$$

where  $d_x$  is the diameter corresponding to  $x\%$  volume on a cumulative size distribution curve.

To determine the porosity of the packed beds, particle density,  $\rho_p$ , and bulk density,  $\rho_b$ , were measured in water and air, respectively. In the next step, porosity was calculated using the formula below (Sahin et al., 2014).

$$\varepsilon = 1 - \frac{\rho_b}{\rho_p} \quad (5)$$

Rheological tests were conducted using an Anton Paar Physica MCR 501 rheometer in a cone-plate system at a temperature of  $20 \pm 0.1$  °C. Since the emulsions under study were non-Newtonian fluids, the Carreau model was applied to describe their rheological properties:

$$\eta = \eta_{\infty} + \frac{\eta_0 - \eta_{\infty}}{[1 + (\lambda \cdot \dot{\gamma})^2]^{\frac{1-n_c}{2}}} \quad (6)$$

where  $\eta_0$  – zero shear viscosity,  $\eta_{\infty}$  – infinite shear viscosity,  $\dot{\gamma}$  – shear rate,  $\lambda$  – time constant, and  $n_c$  – power law exponent. The rheological parameters in Equation (6) were calculated in the Rheoplus software (Anton Paar).

The type of emulsions obtained (o/w or w/o) was evaluated with the dilution method, which is based on the principle that an emulsion can only be diluted with a liquid that is perfectly miscible with the continuous phase. The possibility of diluting the resulting emulsion with the aqueous and oil phases was examined.

## 2.2. Emulsions and dynamic membrane

The aqueous phase of the tested emulsions was a 3 % m/m NaCl solution (POCH). The oil phase was SHELLSOL D100 (viscosity at 20 °C – 0.0029 Pa·s) consisting of 5% v/v Span 80 (Fluka Analytical). The phases were mixed together at a volume ratio of 1:1.

The dynamic membrane was prepared by packing unconsolidated porous media on the support sieve. Glass beads GB (Firma Handlowa Eulalia Bober), polypropylene particles PP (Moplen HP456J, Basell Orlen Polyolefins), polyethylene particles PE (Malen E FABS 23-D022, Basell Orlen Polyolefins) and polyvinyl chloride particles PVC (Polanvil S-58, Anwil) were used in this study. Plain dutch weave 165x1400 (ANN-FILTERS POLAND, warp diameter 0.06 mm, weft diameter 0.04 mm) was used as a support sieve. The particles were sieved using a Retsch AS 200 sieve shaker (100 µm and 150 µm mesh size). As a result, beds of uniform grain size were obtained. The microscopic images of the beds after sieving are shown in Figure 1. The parameters of the packed beds used in the study are listed in Table 1.

Table 1. Characteristics of packed beds

|     | $\varepsilon$ | $d_{32}$ [µm] | $d_{43}$ [µm] | $\delta$ [-] | $C$ [-] |
|-----|---------------|---------------|---------------|--------------|---------|
| PVC | 0.58          | 134           | 140           | 0.62         | 0.51    |
| PP  | 0.52          | 174           | 181           | 0.55         | 0.39    |
| PE  | 0.43          | 135           | 140           | 0.47         | 0.39    |
| GB  | 0.37          | 129           | 133           | 0.35         | 1.00    |

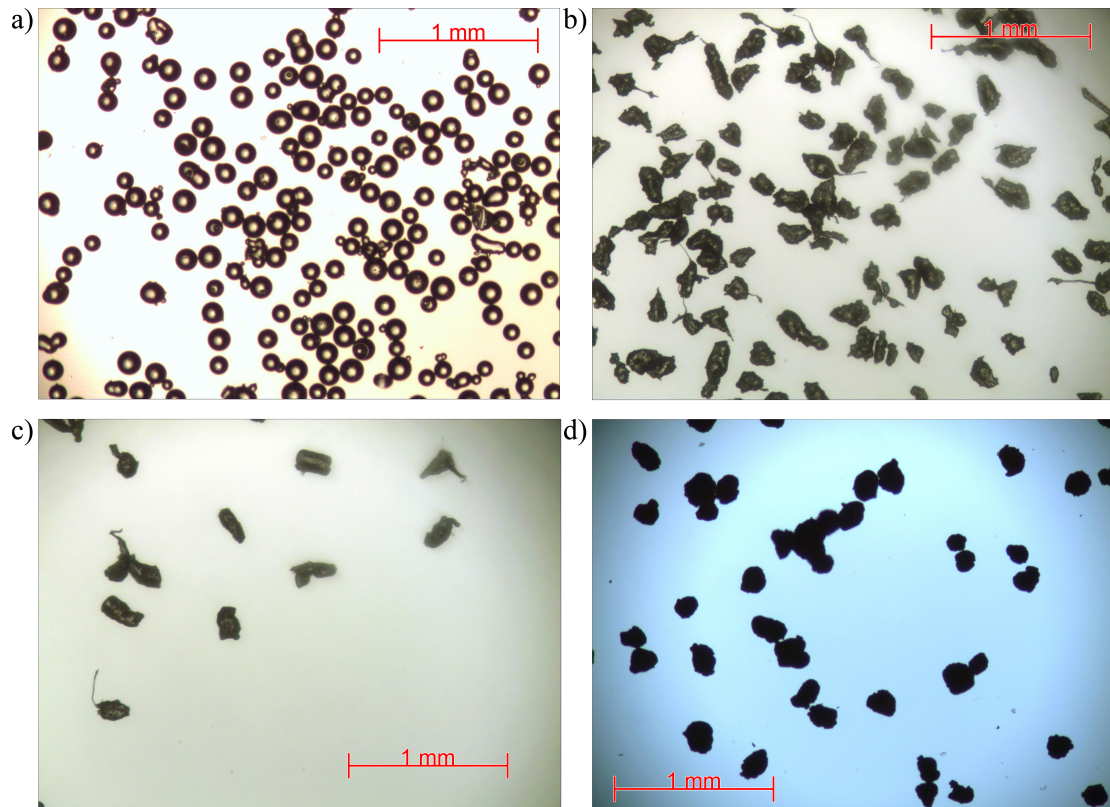


Fig. 1. Porous media: glass beads GB (a), polyethylene particles PE (b), polypropylene particles PP (c), poly(vinyl chloride) particles PVC (d)

### 2.3. Emulsification setup

Experiments were carried out using the in-house designed pressure mixer shown in Figure 2. The components of the emulsion were placed in the tank and mixed by means of an FBT impeller for 300 s. Rotational movement was achieved using an Aquael FAN Plus motor and a magnet placed on the shaft. Mixing produced a premix with the droplet diameter  $d_{32}$  of approximately  $7.3 \pm 1.7 \mu\text{m}$ , and the span of 1.2, which was then passed through a dynamic membrane located in the nozzle. The emulsion flowing out of the nozzle went into a beaker placed on a Radwag WTC 200 balance (reading accuracy: 0.001 g). The increase of mass over time was recorded using the R-Lab software to obtain the mass flow rate,  $G$ . On this basis, it was possible to calculate the superficial velocity defined as (Nazir et al., 2013):

$$v_0 = \frac{G}{\rho \cdot A} = \frac{J}{\varepsilon} \quad (7)$$

where  $A$  – nozzle cross-sectional area,  $\rho$  – emulsion density and  $J$  – flux across the packed bed. This allowed the determination of the modified Reynolds number at the nozzle inlet calculated from the equation:

$$\text{Re} = \frac{v_0 \cdot d_p \cdot \rho}{(1 - \varepsilon) \cdot \eta} \quad (8)$$

where  $d_p$  – particle diameter. The shear rates occurring during the process were also determined from the equation:

$$\dot{\gamma} = \frac{8 \cdot v_0}{d_v \cdot \varepsilon} \quad (9)$$

where  $d_v$  is the interstitial void diameter defined as (Nazir et al., 2013):

$$d_v = \frac{4 \cdot \varepsilon}{\frac{6}{d_p} \cdot (1 - \varepsilon)} \quad (10)$$



Pressure in the tank was measured with a Temat EMS-20L electronic manometer with a measuring range of 0÷690 kPa (accuracy class: 0.6).

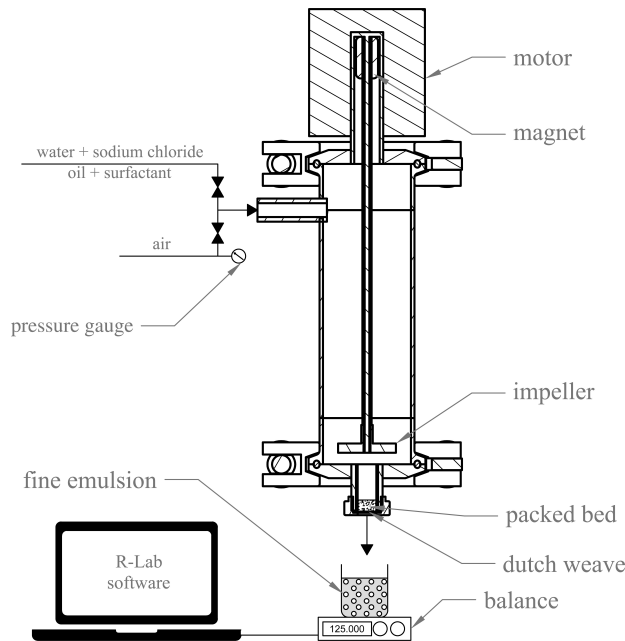


Fig. 2. Experimental setup

#### **2.4. Statistics**

To verify the significance of differences between the test results, an analysis of variance was performed for a single classification using the Statistica 13 software (TIBCO Software Inc.). Each time, the analysis was preceded by checking the assumptions, i.e. verification whether the variables studied had a normal distribution (by means of the Shapiro-Wilk test) and whether these distributions shared the same variance (using the Brown-Forsythe test). Where the analysis of variance showed significant differences between the means considered, Duncan's multiple range test was performed to determine which means differed in a statistically significant manner. All the hypotheses were verified at a significance level of 0.05 (Stanisz, 2007).

### **3. RESULTS AND DISCUSSION**

#### **3.1. Effect of using different packed beds**

A series of experiments were carried out in order to compare the parameters of emulsions obtained using dynamic membranes composed of different particles. Hydrophilic glass beads and hydrophobic particles of polyethylene, polypropylene and poly(vinyl chloride) were used as packed beds. In each test, the dynamic membrane consisted of a 5 mm bed. The applied pressure varied from 250 to 600 kPa. In each case, water-in-oil emulsions were obtained. Table 2 summarises the droplet diameters and flux values across the packed bed depending on the applied pressure. The results of Duncan's test comparing the significance of differences between the premix and emulsions obtained with the dynamic membrane were also presented (the differences were considered significant when  $p < 0.05$ ). The droplet diameters obtained at the lower applied pressures (up to 3.5 bar for GB and PE, and up to 3 bar for PVC and PP) were not statistically different from those of the premix. At higher pressures, though, a reduction in droplet size was observed.

Table 2. Diameters ( $d_{32}$ ) and fluxes obtained across the packed bed as a function of applied pressure and results of the significance test for differences between the fine and premix emulsions

| $P$ [bar] | $d_{32}$ [ $\mu\text{m}$ ] |      |      |      | $J$ [ $\text{m}^3/(\text{m}^2\text{s})$ ] |        |        |        | Duncan's test, $p$ |      |      |      |
|-----------|----------------------------|------|------|------|---|--------|--------|--------|--------------------|------|------|------|
|           | PVC                        | PP   | GB   | PE   | PVC                                       | PP     | GB     | PE     | PVC                | PP   | GB   | PE   |
| 6         | 4.47                       | 4.63 | 5.16 |      | 0.2040                                    | 0.2415 | 0.0859 |        | $p < 0.05$         |      |      |      |
| 5.5       | 5.19                       | 4.83 | 5.57 | 5.17 | 0.1895                                    | 0.2214 | 0.0801 | 0.0408 | $p < 0.05$         |      |      |      |
| 5         | 5.18                       | 5.01 | 5.02 | 6.05 | 0.1746                                    | 0.1984 | 0.0714 | 0.0380 | $p < 0.05$         |      |      |      |
| 4.5       | 5.38                       | 5.74 | 6.13 | 5.65 | 0.1602                                    | 0.1773 | 0.0645 | 0.0351 | $p < 0.05$         |      |      |      |
| 4         | 5.74                       | 5.6  | 5.39 | 6.17 | 0.1445                                    | 0.1550 | 0.0573 | 0.0321 | $p < 0.05$         |      |      |      |
| 3.5       | 4.88                       | 5.83 | 7.59 | 6.34 | 0.1296                                    | 0.1343 | 0.0502 | 0.0293 | $p < 0.05$         | 0.08 | 0.58 |      |
| 3         | 6.43                       | 6.46 | 6.44 | 6.53 | 0.1146                                    | 0.1125 | 0.0429 | 0.0264 | 0.14               | 0.16 | 0.14 | 0.11 |
| 2.5       | 7.17                       | 7.31 |      |      | 0.0995                                    | 0.0914 |        |        | 0.75               | 0.97 |      |      |

It can be noted that the PVC and PP beds led to the achievement of considerably higher efficiencies than the GB and PE beds across the entire range of applied pressure. All the fluxes are considerably higher than those reported by Suzuki et al. (1998) during premix membrane emulsification with a PTFE membrane for a w/o emulsion (e.g.  $0.002 \text{ m}^3/(\text{m}^2 \cdot \text{s})$  for the pressure of 500 kPa). For the PVC and PP beds, the flux values obtained were comparable to those recorded by Nazir et al. (2013) for dynamic membranes consisting of glass microbeads in o/w emulsions (e.g.  $0.177 \text{ m}^3/(\text{m}^2\text{s})$  for the pressure of 500 kPa).

Nazir et al.'s studies (Nazir et al., 2013) show that the distribution of droplet diameters in o/w emulsions formed during the flow of fluid in the spontaneous droplet forming region ( $\text{Re} < 40$ ) differed from the distribution of droplet diameters in emulsions obtained in the inertial droplet break-up region. To determine the range of flow during the passing through the beds used in this study, the values of the Reynolds number were calculated from Equation (8). The premix viscosity curve in Fig. 3 shows that it is a shear-thinning fluid, and the viscosity starts to stabilise at a constant level in the high shear-rate range. The range of shear rates estimated from Equation (9) which was observed as the emulsion was passed through the membrane, varied from 12,500 to 63,000  $1/\text{s}$ . Very high shear rates observed when passing the premix through the membrane provide grounds to assume that infinite shear viscosity,  $\eta_\infty$ , can be used for calculating the Reynolds number. The value of infinite shear viscosity was determined by approximating the viscosity

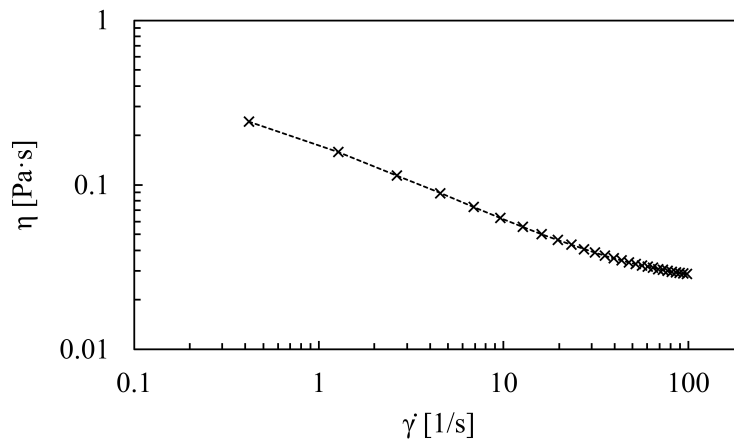


Fig. 3. Premix viscosity curve

curve with the Carreau model (Eq. (6)), which yielded the following model parameters:  $\eta_0 = 0.2690$  Pa·s,  $\eta_\infty = 0.0185$  Pa·s,  $\lambda = 1.639$  s,  $n_c = 0.363$ .

Based on the equation  $d_{32} = f(\text{Re})$  shown in Fig. 4 the flow of emulsion through the bed of PP and PVC particles occurred in the range of considerably higher Reynolds numbers, which should be attributed to their greater porosity, translating into the possibility of obtaining greater flux values across the packed bed at the same pressure levels.

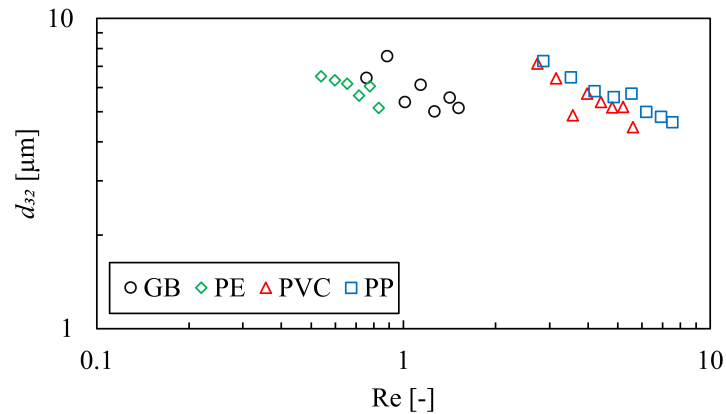


Fig. 4. Droplet diameter,  $d_{32}$ , as function of Reynolds number, Re

In order to assess the homogeneity of the obtained emulsions, the droplet span,  $\delta$ , was calculated and presented as a function of the Reynolds number, Re, in Fig. 5. The emulsions produced with the use of GB and PE membranes are characterized by higher monodispersity compared to the emulsions prepared using PP and PVC membranes.

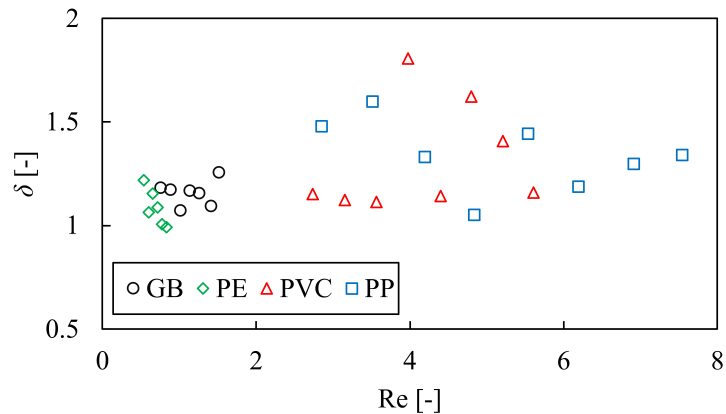


Fig. 5. Droplet span,  $\delta$ , as function of Reynolds number, Re

The differences in droplet span values can be explained by the different ranges of Reynolds numbers at which the experiments were performed. For the GB and PE membranes, the emulsification process was carried out in the range of Re numbers from 0.4 to 1.1, which corresponds to the spontaneous droplet forming region. For the membranes made of PP and PVC, the range of Re numbers was between 2 and 6.2, indicating that the flow would be affected by inertial forces. Research on the preparation of emulsions using both membrane-based (Nazir et al., 2013) and other methods (McClements, 2004) indicates that polydisperse emulsions are formed in the turbulent flow range. Consequently, the formation of emulsions characterized by a wider droplet distribution with the use of PP and PVC membranes can be linked to the impact of inertial forces on the emulsification process.

Microscopic observations of emulsions prepared using a dynamic glass membrane revealed the presence of areas dominated by the aqueous phase (Fig. 6). The images provide evidence to conclude that as the

premix flowed through the glass membrane, some of the droplets disintegrated and some coalesced. This undesirable phenomenon can be associated with the hydrophilic properties of the glass bead surface. Droplets of w/o emulsions that come into contact with a hydrophilic surface are very prone to coalescence.

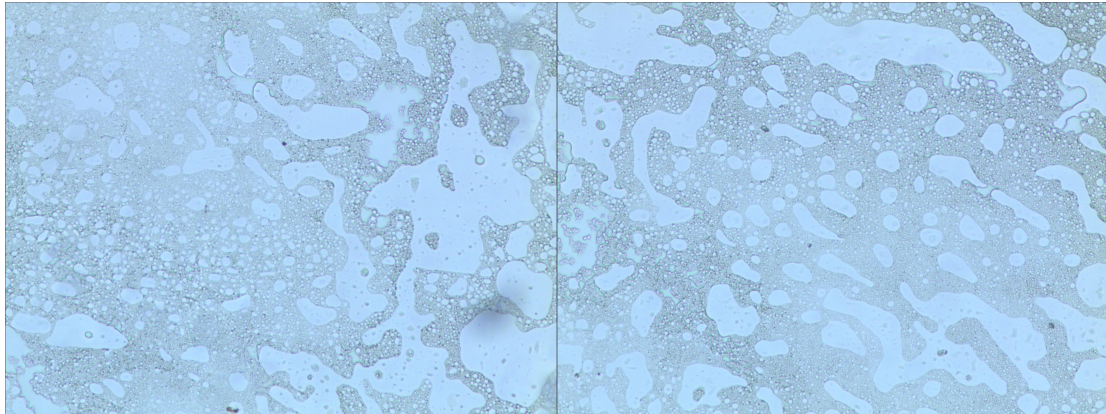


Fig. 6. Adverse phenomenon occurring in emulsions formed with a dynamic glass membrane

### 3.2. Multistage premix membrane emulsification

A comparison of the dynamic membranes used in this study shows that the emulsion with the smallest droplet diameter was obtained by using polypropylene particles in membrane design. The flow of emulsion through the polypropylene membranes was also associated with the highest fluid fluxes. For these reasons, this membrane was used for multistage premix membrane emulsification tests. A membrane made of glass beads was also analyzed for comparison purposes. The emulsions were passed through the membranes four times, at a set pressure of 6 bar. Figure 7 shows the effect of the number of passes on mean droplet diameters. As can be seen, the values of the Sauter mean diameter obtained for a given pass are similar for both beds, so de Brouckere mean diameters were also calculated, as they reflect the presence of relatively large droplets in the emulsion better. The differences between the beds are very minor. Duncan's test shows that they are statistically insignificant.

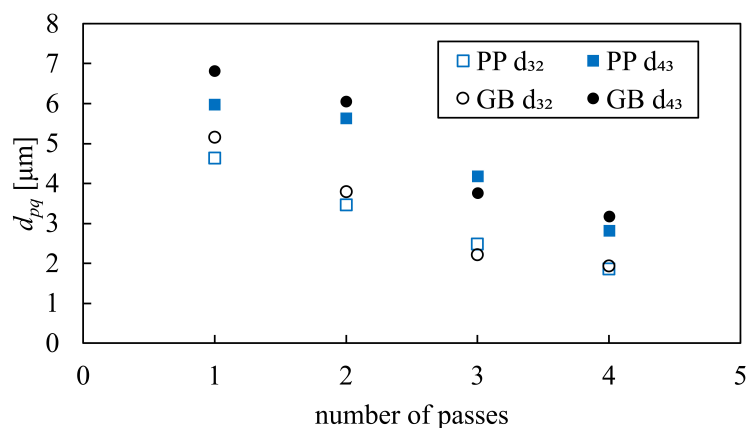


Fig. 7. Effect of the number of passes on the mean diameters of emulsion droplets

To assess the homogeneity of droplet distribution, the span values were calculated. They are shown as a function of the number of passes in Figure 8. The subsequent passes were found to increase the  $\delta$  value, which demonstrates that more polydisperse emulsions were formed. For the third and fourth passes, the emulsions obtained with the glass membrane were characterized by a wider distribution of droplet diameters compared to the emulsions obtained using the polypropylene membrane. After the fourth pass, the differences in span values obtained for different beds were found to be statistically significant.

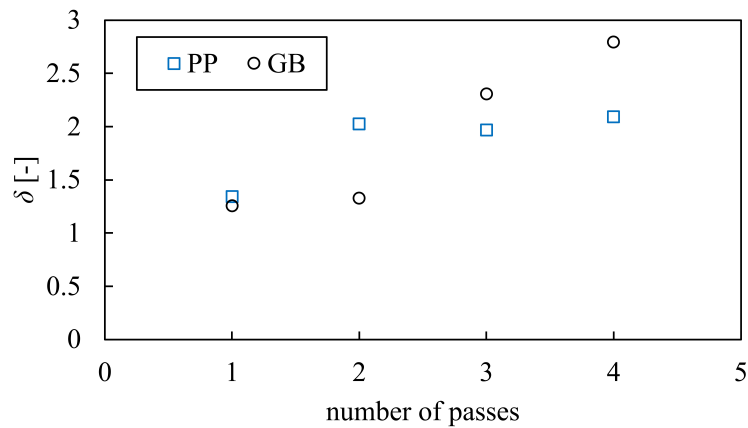


Fig. 8. Effect of the number of passes on the droplet span

In addition to droplet diameter measurements, the emulsions obtained by several passes through a dynamic membrane were also subjected to rheological tests. Pal's studies (Pal, 1996) show that the rheological characteristics of emulsions are closely linked to droplet size, and that a decrease in droplet size leads to a major increase in emulsion viscosity. Figure 9 presents the viscosity curves plotted for the premix and emulsions obtained by repeated passes through the dynamic membrane. The resulting emulsions are non-Newtonian shear-thinning fluids. By passing the premix through the dynamic membrane, the viscosity of the emulsion increased significantly. Subsequent passes also led to an increase in viscosity. The differences between the emulsions were particularly noticeable at low shear stresses. Slightly higher viscosity values were observed in the emulsion prepared using a polypropylene membrane, compared to the emulsion obtained using a glass membrane.

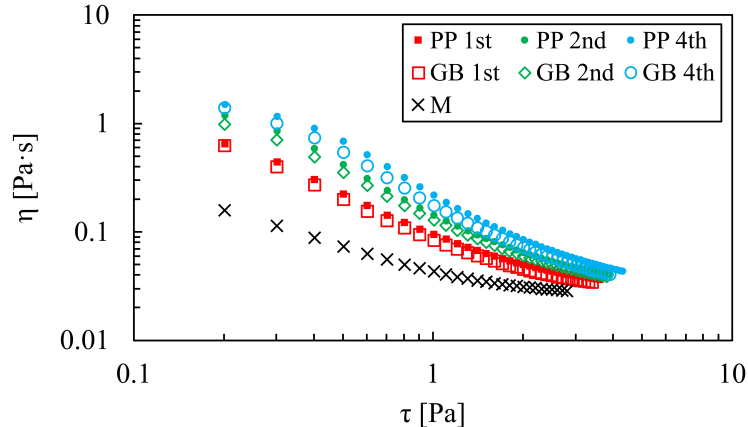


Fig. 9. Viscosity curve for the premix and emulsions obtained by repeated passes through the dynamic membrane

#### 4. CONCLUSIONS

Tests were conducted to determine the suitability of dynamic membranes composed of polypropylene, poly(vinyl chloride) and polyethylene particles of equal grain size for the emulsification of a w/o premix. The dynamic membranes used in the tests were found to reduce the premix droplet diameters when an appropriate pressure level was reached. More porous beds resulted in higher fluxes across the packed bed at the same applied pressure, but the emulsions obtained in this manner were characterised by a wider distribution of droplet diameters. A glass (hydrophilic) membrane also enabled the formation of a water-in-oil emulsion, though the membrane also caused some of the water droplets to coalesce. For the dynamic



polypropylene membrane, the process of multistage premix membrane emulsification was performed. The emulsions obtained after each pass were characterized by steadily decreasing droplet diameter and higher viscosity, but also increasing polydispersity.

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## SYMBOLS

|          |   |
|----------|---|
| $A$      | cross-sectional area, $\text{m}^2$        |
| $C$      | circularity                               |
| $d$      | diameter, m                               |
| $d_p$    | particle diameter, m                      |
| $d_{pq}$ | mean diameter, m                          |
| $d_v$    | pore diameter, m                          |
| $d_{32}$ | Sauter diameter                           |
| $d_{43}$ | de Brouckere diameter                     |
| $d_z$    | circle equivalent diameter, m             |
| $G$      | mass flow rate, kg/s                      |
| $J$      | flux, $\text{m}^3/(\text{m}^2 \text{ s})$ |
| $n_c$    | power law exponent                        |
| $O$      | projection perimeter, m                   |
| $P$      | applied pressure, Pa                      |
| $p$      | $p$ -value                                |
| Re       | Reynolds number                           |
| $S$      | projection area, $\text{m}^2$             |
| $v_0$    | superficial velocity, m/s                 |

### *Greek symbols*

|               |                                 |
|---------------|---------------------------------|
| $\gamma$      | shear rate, 1/s                 |
| $\delta$      | span                            |
| $\varepsilon$ | porosity                        |
| $\eta$        | viscosity, Pa s                 |
| $\eta_0$      | zero shear viscosity, Pa s      |
| $\eta_\infty$ | infinite shear viscosity, Pa s  |
| $\lambda$     | time constant, s                |
| $\rho$        | density, $\text{kg}/\text{m}^3$ |
| $\tau$        | shear stress, Pa                |

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