

## Interfacial Tension of Some Newtonian and non-Newtonian Fluids by the Drop-Weight Method

R. Gupta, R. K. Wanchoo,\* and A. Bansal

Department of Chemical Engineering & Technology,  
Panjab University, Chandigarh, 160 014, India

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Experimental data on interfacial tension of 42 binary liquid-liquid systems using water, aqueous glycerol, aqueous polymeric solutions of polyvinylpyrrolidone (PVP), poly(ethylene oxide) (PEO), carboxymethyl cellulose (CMC) and polyacrylamide (PAM) as continuous phase, and carbon tetrachloride, chlorobenzene, bromobenzene, nitrobenzene, nitrotoluene and ethyl chloroacetate as dispersed phase at 20 °C are reported. The interfacial tension has been measured using the drop-weight method at different compositions of continuous phases.

*Key words:*

Drop weight method, interfacial tension, Newtonian and non-Newtonian liquids

### Introduction

Contacting of one liquid phase with another is a common phenomenon in chemical engineering applications. The objective of such an operation may be the transfer of heat or mass, but momentum transfer will always occur. Examples of such processes include liquid-liquid extraction, direct heat transfer, processing of food, wastewater treatment such as activated sludge process, production of paints and detergents etc. Studies on motion of a dispersed phase through an immiscible liquid are of immense industrial and academic interests.

In all direct contact transfer processes, the two-phase interface plays a significant role in controlling the size, shape and the terminal velocity of the dispersed phase liquid drop, and hence the transfer efficiency of the system. Although, interfaces are at most only a few molecules thick, they are present in many systems, where they are no less important than the phases they enclose. It is therefore important in many industrial applications to know the interfacial tension of these phases.<sup>1</sup> For many years, surface chemists have occasionally measured surface tension of some liquids (generally water) in contact with vapors of immiscible, more volatile liquids at constant temperatures and varying pressures primarily to deduce adsorption isotherms.<sup>2–6</sup> Some data on interfacial tension using water and organic liquid combinations have also been reported.<sup>7,8</sup> These measurements are limited to specific combinations of substances and in most cases, over a narrow temperature range. A captive drop instrument for measuring surface and interfacial tension at high temperatures

and pressures has been successfully used by Schramm.<sup>9</sup> Besides, many other techniques like the drop image processing method,<sup>10</sup> axisymmetric drop technique<sup>11</sup> and method of touching drops<sup>12</sup> which are available in the literature for measuring the surface and interfacial tension of liquids, the drop-weight method described by Harkins and Brown<sup>13</sup> finds a special significance.<sup>14–16</sup> However, most of these studies are focused on interfacial tension measurements for Newtonian-Newtonian liquid systems only. In the case of bioprocess separation and liquid-liquid extraction systems, we usually encounter a combination of Newtonian-non-Newtonian immiscible liquids in contact with each other. In such systems, interfacial tension plays an important role that decides the shape and size of dispersed phase liquid. To the best of our knowledge, not much interfacial data are available on Newtonian-non-Newtonian systems in the literature. Recently, Wanchoo *et al.*<sup>17</sup> have reported interfacial tension data on a few systems involving the formation of Newtonian liquid drop in an immiscible non-Newtonian continuous phase liquid.

According to Harkins and Brown,<sup>13</sup> the mass of the cylindrical drop formed at the tip of the capillary is always less than the mass of the rising liquid in the capillary itself and this fraction of the drop which actually falls has been correlated as a function of  $r/V_d^{1/3}$  where  $r$  is the radius of the capillary and  $V_d$  is the volume of one drop. Accordingly, if this drop is formed in another liquid, the interfacial tension ( ${}_1\gamma_2$ ) between the two immiscible liquids can be correlated as<sup>13</sup>

$${}_1\gamma_2 = \frac{V_d(\rho_1 - \rho_2)g}{2\pi r f(r/V_d^{1/3})} \quad (1)$$

\* Author for correspondence: e-mail: wanchoo@pu.ac.in;  
Fax : +91-01722779173; Tel.: +91-0172-2534933

or

$${}_1\gamma_2 = \frac{V_d(\rho_1 - \rho_2)gF}{r} \quad (2)$$

where,

$$F = \frac{1}{2\pi f (r/V_d^{1/3})}$$

where  $\rho_1$  and  $\rho_2$  are the densities of the two liquids and  $g$  is the acceleration due to gravity.

The correction factor  $F$  has been determined by Harkins and Brown<sup>13</sup> and the data has been correlated in the form of empirical equations by various authors.<sup>18,19</sup> For best results, using the drop weight method, the tip of the capillary should be so selected that  $r/V_d^{1/3}$  lies in the range of 0.6 to 1.2. The uncertainty of this method<sup>20</sup> is reported to be less than 0.1 %. Due to simplicity and reasonable accuracy of the drop weight method, we have used this method to measure interfacial tension for the systems of our own interest involving a combination of Newtonian dispersed phase and non-Newtonian continuous phase.

## Materials and methods

### Determination of interfacial tension

The schematic diagram of the drop – weight method apparatus used in this study is given in Fig. 1. The main components of the apparatus consisted of a glass receiver (1), a specially designed Y-shaped assembly having capillary (2) of known diameter for drop formation, a small funnel (5) used for prefilling of a heavier phase and a graduated pipette attached at socket (3). Capillaries of different diameters were used for different systems such that  $r/V_d^{1/3}$  would always lie in the range of 0.6 to 1.2.

**Procedure.** The set up (Fig. 1) was used to determine the interfacial tension between lighter liquid i.e. distilled water, glycerol (20 % (w/v) and 25 % (w/v)), aqueous polymeric solutions, and heavier liquid (carbon tetrachloride, chlorobenzene, bromobenzene, nitrobenzene, nitrotoluene, and ethyl chloroacetate) at a constant temperature. The drop weight apparatus was calibrated using distilled water as lighter liquid in the receiver, and different organic liquids (hexane, chlorobenzene, carbon tetrachloride, benzene) as heavier liquid in the capillary. The values of interfacial tension so determined, were compared with the data available in the literature<sup>24</sup> and the % deviation was calculated. These values are given in Table 4. While carrying out the experiment, heavier phase liquid was taken in the graduated pipette and fitted in the left joint of the Y-assembly. A stopper was used to block the capillary from the bot-

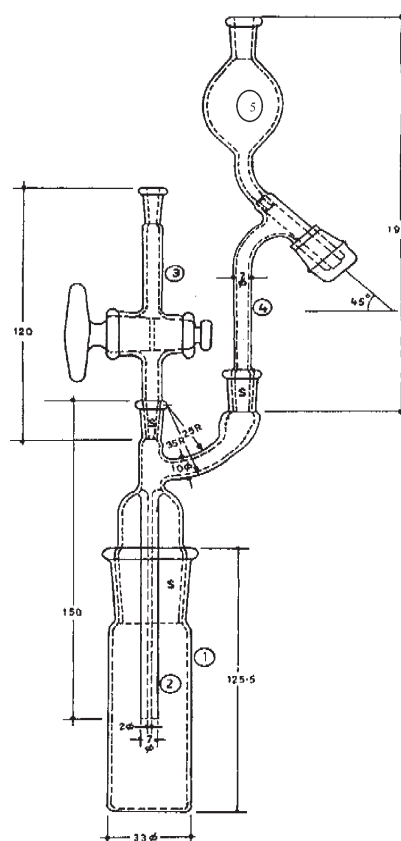


Fig. 1 – Drop-weight apparatus wherein parts 1, 2, 3, 4 and 5 are receiver, capillary, joint for fixing calibrated pipette carrying heavier liquid for drop formation, heavier liquid inlet, and reservoir for pre-filling of heavier liquid respectively. S represents standard glass joint. All dimensions are in mm.

tom. Keeping the left joint of the Y-assembly in closed position allowed the heavier phase liquid to fill the capillary by opening the right hand side stopcock. Then the left side stopcock slowly opened, allowing the heavier phase liquid to fill in the space above the capillary, and expelling any air if present in the limb of the Y-assembly. Closed both the stopcocks and placed this assembly into a glass receiver containing lighter phase liquid. The height of the lighter phase liquid should be such that the capillary is completely dipped in the lighter phase liquid. The initial level of the heavier liquid in the graduated pipette was then noted.

The complete set up was kept in a glass vessel with arrangement for circulating water around the assembly maintained at the desired temperature of  $20\text{ }^\circ\text{C} \pm 0.1$  using Julabo constant temperature circulating bath. The opening of the stopcock below the graduated pipette was adjusted in such a manner that it took more than 3 minutes for the formation of one single drop at the tip of the capillary. In this manner, a large number of drops were formed and the volume of drops was measured after noting the initial and final levels of the heavier liquid in the pipette. The volume of a single drop was thus cal-

culated corresponding to which the correction factor  $F$  was determined from the table given by Harkins and Brown,<sup>13</sup> where direct values of  $F$  were not available, correlations proposed by Wilkinson and Kidwell<sup>19</sup> were used. The interfacial tension between the two liquids was thus determined using eq. (2). The same procedure was then repeated to calculate the interfacial tension between different organic liquids (Table 1) and aqueous polymeric solutions, which formed a combination of Newtonian – non-Newtonian systems (Table 2).

During experimentation, care was taken to ensure that all joints were leak proof. In each case sufficient equilibrating (> 15 min) time was given so that the receiver and the capillary liquid attained the desired temperature before the start of the experiment.

### Determination of density

The densities of pure heavier liquids (carbon tetrachloride, chlorobenzene, bromobenzene, nitrobenzene, nitrotoluene, and ethyl chloroacetate), all Merck reagent grade chemicals, were measured using density meter (DMA 48, Anton and Paar) at  $20 \pm 0.1$  °C and the values are given in Table 1. The purity of the chemicals was checked by comparing the experimental value of density with literature values.<sup>23</sup>

Table 1 – Density,  $\rho_d$ , of heavier liquids (dispersed phase) at  $\vartheta$ , °C =  $20.0 \pm 0.1$

Liquid	$\rho_d/\text{kg m}^{-3}$	$\rho_{lit}^{23}/\text{kg m}^{-3}$
carbon tetrachloride	1592.0	1591.2
bromobenzene	1495.0	1495.0
nitrobenzene	1203.3	1203.7
nitrotoluene	1162.0	1162.9
ethyl chloroacetate	1150.0	1150.0
chlorobenzene	1106.3	1106.1

### Characterization and rheology of polymers

Different aqueous polymeric solutions of polyvinylpyrrolidone (PVP), poly(ethylene oxide) (PEO), carboxy methyl cellulose (CMC), polyacrylamide (low viscous) (PAMlv) and polyacrylamide (high viscous) (PAMhv) were prepared in distilled water and were characterized using the viscometric method.<sup>28</sup> Viscosity average molecular mass ( $M_V$ ) for each polymer was based on measured values of intrinsic viscosity. The values of viscosity average molecular mass,  $M_V$ , obtained for each polymer and density of their aqueous solutions at 20 °C are tabulated in Table 2. The rheological parameters of all polymeric solutions were evaluated using cone

and plate rheometer (RT-20, Haake, Germany; Type C 60/1 Ti, Plate diameter 60 mm; Cone angle 1°; Truncation 0.053 mm) at 20 °C and are also given in Table 2. Errors for all measured quantities have been estimated using Mickley's method<sup>22</sup> and the values are tabulated in Table 3.

Table 2 – Characterization, rheological parameters  $K$ ,  $n$  and density,  $\rho_c$ , of lighter liquids (continuous phase) as a function of concentration  $\gamma$  at  $\vartheta$ , °C =  $20.0 \pm 0.1$

Liquid	100 $\gamma/\text{g cm}^{-3}$	$K/\text{Pa s}^n$	$n$	$\rho_c/\text{kg m}^{-3}$
water	100	$1.005 \cdot 10^{-3}$	1	997.9
glycerol	20	$1.886 \cdot 10^{-3}$	1	1037.4
	25	$1.977 \cdot 10^{-3}$	1	1070.0
PVP* $M_V = 2.7 \cdot 10^4$	0.50	$1.071 \cdot 10^{-3}$	1	999.0
	0.75	$1.147 \cdot 10^{-3}$	1	999.5
	1.00	$1.224 \cdot 10^{-3}$	1	1000.3
	2.00	$1.358 \cdot 10^{-3}$	1	1003.6
	3.00	$1.566 \cdot 10^{-3}$	1	1005.7
PEO* $M_V = 3.0 \cdot 10^5$	4.00	$1.915 \cdot 10^{-3}$	1	1007.8
	0.80	$4.085 \cdot 10^{-3}$	1	998.5
	1.60	$14 \cdot 10^{-3}$	1	1000.0
CMC* $M_V = 1.0 \cdot 10^5$	3.20	$29.834 \cdot 10^{-3}$	1	1001.6
	0.20	$56.88 \cdot 10^{-3}$	0.80	999.40
	0.50	$330 \cdot 10^{-3}$	0.72	999.85
PAM (lv)* $M_V = 3.6 \cdot 10^6$	1.00	$1486 \cdot 10^{-3}$	0.64	1001.9
	0.01	$0.982 \cdot 10^{-3}$	1	997.8
	0.05	$2.105 \cdot 10^{-3}$	1	998.0
PAM (hv)* $M_V = 8.7 \cdot 10^6$	0.10	$7.657 \cdot 10^{-3}$	0.87	998.1
	0.50	$424.5 \cdot 10^{-3}$	0.54	1020.0
	0.05	$63.967 \cdot 10^{-3}$	0.62	996.9
	0.10	$275.233 \cdot 10^{-3}$	0.43	1000.7

\*PVP is polyvinylpyrrolidone; PEO is poly(ethylene oxide); CMC is carboxymethyl cellulose; PAM(lv) is polyacrylamide (low viscous); PAM(hv) is polyacrylamide (high viscous)

\*\* $M_V$  denotes viscosity average molecular mass

Table 3 – Error in the measurement of experimental quantities

Quantity	Maximum error
drop diameter	$2r \pm 0.01 \cdot 10^{-3} \text{ m}$
drop volume	$V_d \pm 0.01 \text{ mL}$
density	$\rho \pm 1 \cdot 10^{-1} \text{ kg m}^{-3}$
interfacial tension	$1\gamma_2 \pm 0.19 \text{ mN m}^{-1}$
temperature	$\vartheta \pm 0.1 \text{ °C}$

## Results

Using the measured values of drop volume ( $V_d$ ), density of heavier phase liquid ( $\rho_1$ ), density of lighter phase liquid ( $\rho_2$ ), capillary radius ( $r$ ) and correction factor ( $F$ ), the interfacial tension ( ${}_1\gamma_2$ ) was obtained using eq. (2). The data is given in Table 5. The accuracy of the results obtained by the present drop-weight apparatus was established by comparing the measured values of interfacial tension of some Newtonian systems with the values available in the literature. As is observed from Table 4, the accuracy of measurement in the present case expressed in terms of MRQE (mean relative quadric error) is 0.014. Where MRQE is

defined as: 
$$\sqrt{\frac{\sum_{i=1}^n \left( \frac{\gamma_{\text{exp}} - \gamma_{\text{lit}}}{\gamma_{\text{exp}}} \right)^2}{N - 1}}$$
,  $N$  is the number of

data points taken. Based on error estimates of various physical quantities, the values of interfacial tension,  ${}_1\gamma_2$  are accurate to within  $\pm 0.19 \text{ mN m}^{-1}$  (Table 3).

Table 4 – Comparison between observed values of interfacial tension  ${}_1\gamma_2$  (water + organic liquid) and the literature values

Organic liquid	$\vartheta/^\circ\text{C}$	$\gamma_{\text{lit}}/24/\text{mN m}^{-1}$	$\gamma_{\text{exp}}/\text{mN m}^{-1}$	$\delta$
hexane	20	51.00	51.44	0.44
chlorobenzene	20	37.41	37.30	-0.11
carbon tetrachloride	20	45.00	45.10	0.10
aniline	20	5.77	5.81	0.04
nitromethane	20	9.66	9.52	-0.14
benzaldehyde	20	15.51	15.98	0.47
carbon tetrachloride	25	43.70	44.03	0.33
benzene	25	34.71	35.01	0.30

$\delta$  = deviation = experimental value – literature value

From the results in Table 1, Table 2 and Table 5, it is observed that:

(a) In case of Newtonian as well as non-Newtonian systems, for the same dispersed phase liquid (heavier phase), as the concentration of lighter phase increases, thereby increasing its density, the interfacial tension decreases.

(b) Further, as the density of heavier liquid decreases, the interfacial tension for the same continuous phase liquid (lighter phase) (Newtonian as well as non-Newtonian) also decreases. With chlorobenzene as an exception.

Table 5 – Values of interfacial tension,  ${}_1\gamma_2$ , ( $\text{mN m}^{-1}$ ) as a function of concentration,  $\gamma$  at  $\vartheta$ ,  $^\circ\text{C} = 20.0 \pm 0.1$

Lighter liquid	100 $\gamma/\text{g cm}^{-3}$	Heavier liquid*					
		$\text{CCl}_4$	CB	BB	NB	NT	ECA
water	100	47.23	36.81	40.67	28.41	27.41	22.91
	20.0	46.56	35.82	39.27	27.14	26.84	22.21
glycerol	25.0	29.94	11.74	24.37	12.27	11.28	11.08
	0.50	47.12	28.45	39.94	28.15	27.04	19.48
PVP**	0.75	39.43	25.65	38.72	26.13	26.87	19.39
	1.00	33.94	13.54	29.13	23.06	21.68	16.84
PEO**	2.00	19.72	12.82	12.15	8.27	8.18	8.03
	3.00	17.24	12.60	12.26	6.67	7.12	6.82
CMC**	4.00	14.68	9.37	9.30	4.98	5.13	5.22
	0.80	38.15	31.04	35.85	24.31	23.31	18.94
PAM(lv)**	1.60	38.02	30.47	32.52	22.58	23.78	14.60
	3.20	35.67	29.43	29.85	19.62	21.19	11.31
PAM(hv)**	0.20	33.52	22.85	32.16	25.65	22.35	19.23
	0.50	32.71	21.84	29.06	21.58	21.23	18.84
PAM(lv)**	1.00	28.52	20.40	25.06	18.55	19.61	16.44
	0.01	38.10	27.40	40.06	27.65	20.60	20.49
PAM(hv)**	0.05	36.22	27.12	32.38	21.37	19.71	11.07
	0.10	39.35	26.80	21.40	16.52	19.24	9.00
PAM(hv)**	0.50	14.52	6.85	14.35	8.75	12.71	9.09
	0.01	36.27	20.41	21.06	13.12	15.07	10.09
PAM(hv)**	0.05	35.94	19.69	20.12	12.77	14.24	8.99
	0.10	18.41	7.62	14.37	9.95	7.76	5.87
PAM(hv)**	0.15	16.87	7.26	13.97	9.11	7.81	5.92
	0.20	15.35	6.88	13.77	9.02	7.31	5.76

\* $\text{CCl}_4$  is carbon tetrachloride, CB is chlorobenzene; BB is bromobenzene; NB is nitrobenzene; NT is nitrotoluene; ECA is ethyl chloroacetate.

\*\*PVP is polyvinylpyrrolidone; PEO is poly(ethylene oxide); CMC is carboxymethyl cellulose; PAM(lv) is polyacrylamide (low viscous); PAM(hv) is polyacrylamide (high viscous)

(c) For the same dispersed phase liquid (heavier phase), with increase in liquid phase viscosity of lighter phase, interfacial tension decreases.

## Discussion

After establishing the accuracy of the inexpensive drop weight method, the interfacial tension between six organic liquids and immiscible Newtonian and non-Newtonian continuous phase liquids

was measured. Combinations of Newtonian and non-Newtonian liquid systems find applications in liquid-liquid extraction, direct contact heat transfer, biological systems etc. To our best knowledge, the values of interfacial tension for such systems are not available in the literature.

Using the experimentally observed values of interfacial tension,  ${}_1\gamma_2$ , given in Table 5, along with density ratio of dispersed to continuous phase liquid (i.e. heavier liquid phase density/lighter liquid phase density =  $\rho_d/\rho_c$ ) (using values given in Table 1 and Table 2), it is observed that, interfacial tension,  ${}_1\gamma_2$ , increases with increase in density ratio, ( $\rho_d/\rho_c$ ) of the two liquids (Fig. 2). However, with chlorobenzene as dispersed phase is an exception. This behavior is observed both in the case of Newtonian-Newtonian systems as well as in the Newtonian-non-Newtonian system. The effect of viscoelasticity can be seen from Fig. 2. It has been observed that for the same value of density ratio, interfacial tension decreases. Similar results were obtained using the values of interfacial tension reported in the literature<sup>25–27</sup> (Fig. 3). The viscosity ratio ( $\mu_d/\mu_c$ ) was calculated using the values of  $\mu_d$  (obtained at a temperature of  $20 \pm 0.1$  °C) and  $\mu_c$ . Using these viscosities, it was observed that the interfacial tension,  ${}_1\gamma_2$ , decreases with increase in viscosity ratio ( $\mu_d/\mu_c$ ) (Fig. 4). A similar observation was made on available literature data<sup>25–27</sup> as well. The regression technique was used to elucidate the effect of ( $\rho_d/\rho_c$ ) and ( $\mu_d/\mu_c$ ) on the interfacial tension using the present data (Fig. 5). The data is represented well by the following mathematical equation.

$${}_1\gamma_2 = 15.6 e^{0.719 \left( \frac{\rho_d}{\rho_c} / \frac{\mu_d}{\mu_c} \right)^{0.33}} \quad \text{with } r^2 = 0.99 \quad (3)$$

within the accuracy of experimental data. Fig. 5 shows that the data available in the literature is also represented by the present eq. (3).

### Conclusion

In the present study, interfacial tension data between some binary systems, including organic liquids as dispersed phase (heavier phase) and Newtonian and non-Newtonian (visco-inelastic and visco-elastic) liquids as continuous phase (lighter phase) have been reported.

For all Newtonian and non-Newtonian systems under study it has been observed that, as the density ratio ( $\rho_d/\rho_c$ ) increases, the interfacial tension ( ${}_1\gamma_2$ ) increases (Figs. 2 and 3). The interfacial tension ( ${}_1\gamma_2$ ) decreases as the viscosity ratio ( $\mu_d/\mu_c$ ) increases (Fig. 4). This combined effect of change in density and viscosity ratio can be represented to within reasonable accuracy by eq. (3).

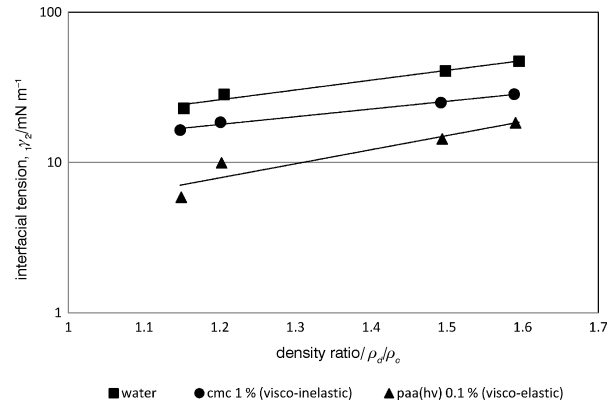


Fig. 2 – Variation of interfacial tension with density ratio (Newtonian, non-Newtonian viscoelastic, non-Newtonian viscoelastic)

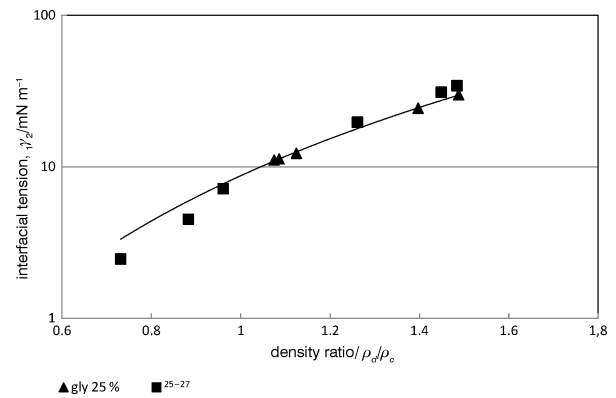


Fig. 3 – Variation of interfacial tension with density ratio

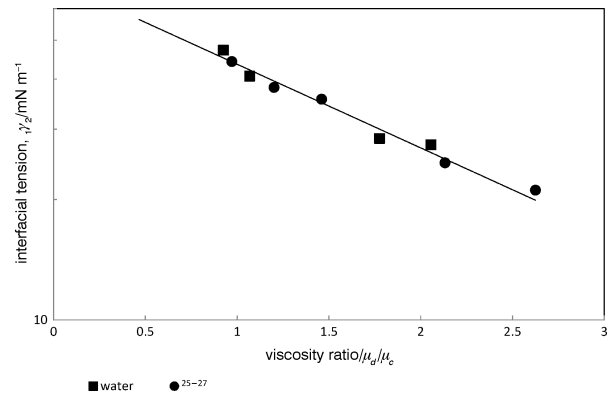


Fig. 4 – Variation of interfacial tension with viscosity ratio

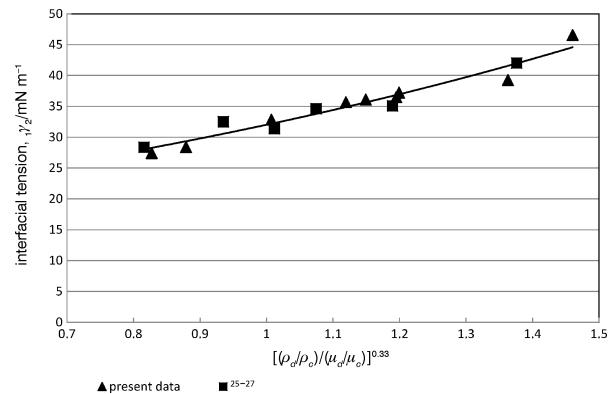


Fig. 5 – Variation of interfacial tension with density ratio and viscosity ratio

**List of symbols**

$F$	– correction factor
$f$	– function
$K$	– flow consistency index, Pa s <sup>n</sup>
$M$	– relative molecular mass
$N$	– number of data points
$n$	– flow behavior index
$r$	– radius, m
$r^2$	– correlation coefficient
$V$	– volume, mL
$\gamma$	– mass concentration, g cm <sup>-3</sup>
${}_1\gamma_2$	– interfacial tension, mN m <sup>-1</sup>
$\delta$	– deviation
$\theta$	– temperature, °C
$\mu$	– dynamic viscosity, Pa s
$\rho$	– density, kg m <sup>-3</sup>

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