

The Effect of Viscosity on the Dynamic Surface Tension Measurements of Printing Inks and Coatings.

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Abstract

Low Dynamic Surface Tension (DST) of printing inks and coatings is one of several very important factors responsible for good printability. The maximum bubble pressure and differential maximum bubble pressure techniques are among the most often used methods to measure the surface tension of printing inks and coatings under different dynamic conditions. Such measurements can be affected by different factors such as: surface properties of liquids and capillaries, probe immersion depth, capillary forces, viscosity and rheology, etc. Some of those factors are considered minor but some play a major role and can be responsible for significant errors. Those factors are briefly discussed in this paper with special emphasis on viscosity. The so-called viscous effect (resistance of fluid against the moving bubble) is responsible for erroneous DST data obtained for viscous liquids. A model system consisting of water/glycerol solutions of different viscosity has been selected to investigate the effect of viscosity on the dynamic surface tension measurements under different dynamic conditions and for different types of capillaries. It was found that the error due to the viscous effect increased with increasing viscosity, increasing capillary radius and decreasing bubble surface age. This error for hydrophobic capillaries was significantly lower than that for hydrophilic probes.

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Introduction

During printing, one deals with a continuous process of bringing together two different surfaces (ink and substrate) into physical contact in the nip of the printing press. A new type of the interface is formed whose properties depend on the surface properties of both ink and substrate. The surface properties of inks are extremely important for achieving good printability. They affect such phenomena at the interface as wetting, spreading, leveling, adhesion and penetration into porous substrates such as paper and board (Adamson and Gast, 1997; Hunter, 2001; Janule, 2001; Schwartz, 1994). A good knowledge and understanding of surface properties of inks and substrates can help in better understanding of the system and predict ink behavior on the press and achieve high quality prints.

It is well known and commonly accepted that in order to achieve good printability, printing inks should have low surface tension (γ). The low surface tension is necessary for good ink lay. Solvent-based inks have inherently low surface tension due to the solvent used e.g. n-propyl alcohol ($\gamma = 23$ mN/m). Water, on the other hand, has high surface tension ($\gamma = 73$ mN/m). However, the surface tension of water-based inks can be lowered to a more appropriate level by addition of surfactants.

There are a number of different experimental methods for measuring surface tension of liquids (Adamson and Gast, 1997; Rosen 1989; Ross and Morrison, 1988). Some of them can be used to measure surface tension under equilibrium conditions (static) while others can be used to measure the surface tension under non-equilibrium conditions – so-called dynamic surface tension (DST). To relate the experimental data on surface tension of printing ink or coating to the printing or coating processes, the data should be acquired under dynamic conditions. Two widely used methods for the DST measurements of liquids are maximum bubble pressure (MBP) and differential maximum bubble pressure (DMBP). Commercial instruments based on the above concepts are available on the market. For instance, MPT1 and MPT2 tensiometers by Lauda Germany and BP2 tensiometer from Kruss, Germany use the MBP concept while Sensadyne tensiometers by Chem Dyne Research Corporation, USA use the DMBP concept.

Though both MBP and DMBP methods appear to be very simple, the interpretation of experimental data acquired with such tensiometers is not trivial. The measurements can be affected by many factors such as capillary surface properties (inside and outside walls), immersion depth and displacement effects, capillary forces acting inside capillary, adsorption processes at the interfaces, viscosity and rheology of liquid, etc. Some of these factors may have only minor effect on the DST and can be neglected under certain circumstances. On the

other hand, other factors such as liquid viscosity and rheological properties may be responsible for the error exceeding a few hundred per cent of the true DST value.

The aim of the present paper is to discuss briefly the effects of some factors on the dynamic surface tension measurements of liquids, using MBP and DMBP methods, with special emphasis on the viscous effect. This is a very important issue for printers and ink and coating manufacturers. The viscous effect can be neglected for measurements performed in low viscosity media such as organic solvents, water, and diluted aqueous solutions. However, the viscosity of liquid printing inks and coatings may be significantly higher than viscosity of water or pure organic solvent and may range from 5 mPas to a few hundreds of mPas. The studies described in this paper were performed using model systems consisting of water/glycerol solutions ranging in viscosity from 1 mPas up to 760 mPas at 22°C. A procedure for the correction of the apparent DST results for a viscous effect was proposed for the DMBP method.

Theory

Basic concept

The concept of measuring dynamic surface tension of liquids using a maximum bubble pressure technique (Fainerman, Miller and Joos, 1994; Mobius and Miller, 1998) is based on the measurement of a pressure required to generate a bubble at the tip of a narrow capillary during controlled gas flow through this capillary immersed in the liquid (ink, coating, solvent, etc.).

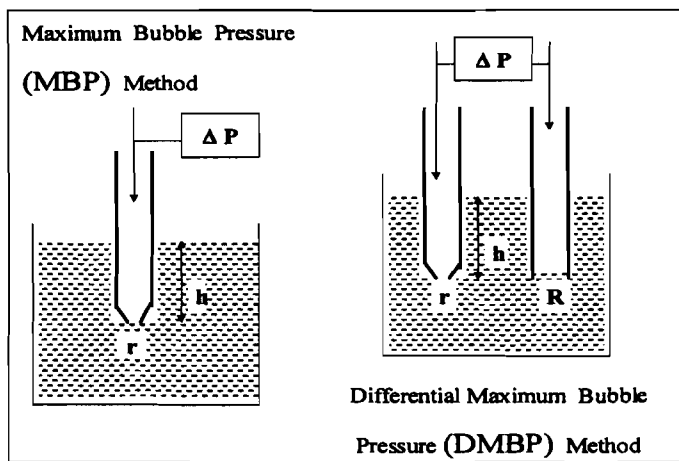


Fig.1. Schematic representation of the Maximum Bubble Pressure (MBP) and Differential Maximum Bubble Pressure (DMBP) methods.

For a single cylindrical capillary (MBP method) of a radius “r” the excess pressure (ΔP) required for this process is (Fainerman, Miller and Joos, 1994; Mobius and Miller, 1998) – see Fig. 1:

$$\Delta P = \frac{2\gamma}{r} + \rho gh \quad (1)$$

where: “ ρ ” is liquid density, “g” is acceleration of gravity and “h” is the immersion depth of the capillary. Eq. (1) takes into account the surface tension of the liquid and hydrostatic pressure only and is applicable to the both systems that are free or contain surface-active agents.

For differential maximum bubble pressure (method schematically presented in Fig. 1) - two capillaries of different radii “r” and “R” immersed at the same depth – by analogy to Eq. (1) and for liquids free of surfactants one can write:

$$\Delta P = \left(\frac{2\gamma}{r} + \rho gh \right) - \left(\frac{2\gamma}{R} + \rho gh \right) \quad (2)$$

or

$$\Delta P = 2\gamma \left(\frac{1}{r} - \frac{1}{R} \right) \quad (3)$$

For liquids containing surfactants, the dynamic conditions at both orifices may be different (e.g. orifices bubbling at different rates) and thus the values of surface tension at the orifices may differ as well. Therefore, one should use general form of Eq. (2):

$$\Delta P = \left(\frac{2\gamma_r}{r} + \rho gh \right) - \left(\frac{2\gamma_R}{R} + \rho gh \right) \quad (4)$$

or

$$\Delta P = \left(\frac{2\gamma_r}{r} - \frac{2\gamma_R}{R} \right) \quad (5)$$

where: “ γ_r ” is a local surface tension at the tip of small orifice and “ γ_R ” is a local value of surface tension at the tip of large orifice. Eqs (4) and (5) can not be

solved analytically because they contain two unknowns. For practical application (used in Sensadyne instruments) it is assumed that $\gamma_r = \gamma_R$.

Corrections

The results of the DST measurements using MBP and DMBP methods depend on the experimental conditions. This is especially true for the systems containing surfactants. Effects of different factors on the accuracy and reproducibility of the results using the MBP technique were analyzed and are described in the literature (Mobius and Miller, 1998; Mysels, 1986, 1989 and 1990). It was found that the value of measured dynamic surface tension can be affected by such factors as: diameter and wettability of a vessel containing liquid, capillary immersion depth, liquid agitation, wettability of capillary, capillary length, etc. Reproducibility of the results depends greatly on the stability of the process of bubble formation and its volume at the capillary tip (Mobius and Miller, 1998; Mysels, 1986, 1989 and 1990). Foaming of the liquid contributes to inaccurate surface tension readings.

All of the above is also true and applicable to some extent to the DMBP method because not all effects will cancel out as differential pressure readings are taken at two different orifices. For example, the assumption that $\gamma_r = \gamma_R$ is true for pure liquids only. For systems that contain surfactants, even if one adjusts gas flow so that the bubbling rate is the same at both orifices, the rate of surface expansion, bubble growth and detachment, etc., are still different at large and small orifices and thus $\gamma_r \neq \gamma_R$. So far there is no method to correct for this difference (Mobius and Miller, 1998).

Immersion depth – displacement effect

Constant immersion depth of the capillary is very crucial especially for the MBP method and when larger capillaries are used. For instance, assuming the capillary diameter $d = 100$ micrometers and inaccuracy of immersion depth equal to one millimeter the corresponding error in the surface tension value would be equal to 0.25 mN/m. For the capillary of $d = 1$ mm, this error would be equal to 2.5 mN/m. To avoid the error due to inaccuracy in immersion depth, one has to control this parameter very precisely. This is easily achievable in the lab but is rather impractical in real life. Another source of this type of error may be a change of liquid level when the bubbles displace some of the liquid (Mysels, 1990). This may become important in the case of small volumes of sample in a narrow vessel.

For an accurate evaluation of hydrostatic pressure, the immersion depth of the bubble apex ($h + r$) should be taken into account (see Fig.1). Thus Eq. (1) should be expressed as:

$$\Delta P = \frac{2\gamma}{r} + \rho g(h+r) \quad (6)$$

By analogy, Eqs (2) and (4) should be rewritten as:

$$\Delta P = 2\gamma \left(\frac{1}{r} - \frac{1}{R} \right) + \rho g(r-R) \quad (7)$$

and

$$\Delta P = \left(\frac{2\gamma_r}{r} - \frac{2\gamma_R}{R} \right) + \rho g(r-R) \quad (8)$$

Using the DMBP method for pure liquids and assuming that the size and shape of bubbles generated at both orifices are independent on surface tension and liquid density, Eq. (7) can be expressed as:

$$\Delta P = a\gamma + b \quad (9)$$

where: $a = 2(1/r - 1/R) = \text{constant}$ and $b = \rho g(r - R) = \text{constant}$.

Surface properties

Surface properties of capillaries and thus wettability by a liquid under investigation can also affect the surface tension measurements (Mobius and Miller, 1998; Mysels, 1990). For hydrophilic (well-wetted) capillary lumen, the capillary forces (suction of liquid) can contribute to the pressure measured by the instrument. If the wettability of capillary lumen is not perfect (contact angle greater than zero) this correction becomes smaller. It may even reverse the sign for very hydrophobic capillaries for which contact angle is greater than 90°. If both lumen of the capillary and outside wall are hydrophobic, the hemispherical bubble formed at the capillary tip can migrate toward the outside of the capillary so that its radius will increase and its value may be uncertain. The ideal capillary should have a hydrophobic lumen and clean hydrophilic outside wall/ This would fix the position of the bubble attachment to the boundary between the hydrophobic lumen and hydrophilic outside wall and also eliminate the problem of ill-defined film of liquid inside the capillary (Fainerman and Miller, 1995; Mobius and Miller, 1998; Mysels, 1990). Such a condition may be difficult to fulfill in practice. For example, surfactant present in the system may adsorb at the capillary wall/solution interface and drastically alter the surface properties of the capillary wall.

Liquid density

Another source of error while using the DMBP method is the procedure of instrument calibration. Water and alcohol are often used for calibration of DMBP instruments. Simplified Eq. (9) can be used for evaluation of the DST only when calibration liquids and samples have the same densities. This is not the case for alcohol and water which differ significantly in density as well as in their surface tension values. The value of error due to differences in density between calibration liquids and a sample will depend on the density difference and combination of capillaries used. The evaluation of this error was done by Schramm (Schramm, 1989). The error increased with increasing capillary radius and density difference between the sample and calibrating liquid. The example calculations for $r = 0.5$ mm and $R = 2.0$ mm and immersion depth $h = 1.5$ cm are presented in Fig. 2 for different sample densities. Using calibration liquids of different surface tension but the same density can reduce this error. At the same time, it is important for the accuracy of the DST measurements that the surface tension span between calibration liquids should be as close as possible to the surface tension of the liquids tested.

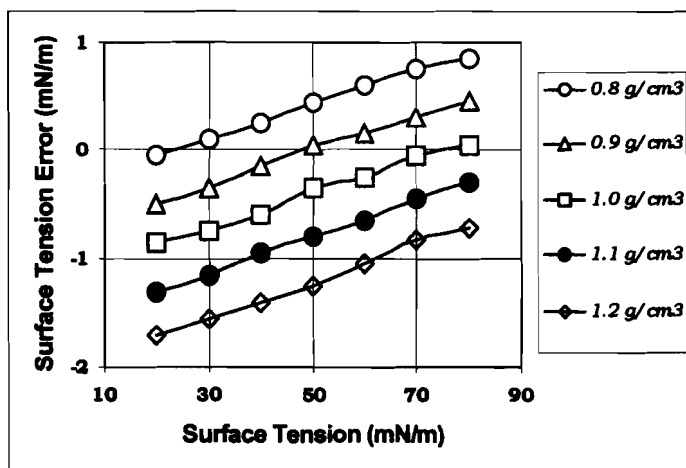


Fig. 2. The errors of the dynamic surface tension measurements vs. the DST for different sample density – DMBP method. Probes diameters: large – 4 mm; small – 1 mm.

Liquids containing surfactants

The measurement of DST by the DMBP method of liquids containing surfactants is even more complicated because for such systems $\gamma_r \neq \gamma_R$ (the rate of surface area increase is ill defined and capillaries may bubble at different rates) (Schramm, 1989; Mysels, 1990). The errors will not cancel out as observed for hydrostatic pressure. No satisfactory solution for this problem has been offered so far. Moreover, the assumption about spherical shape of the bubbles is valid only for very narrow capillaries. For large capillaries, the deviation of the bubble shape from the spherical shape should be also taken into account.

Viscous effect

In the simplified description of both MBP and DMBP methods presented above, all hydrodynamic effects (Δp) have been neglected. In fact, Eqs (1) and (2) should be written, respectively as (Fainerman, Miller and Joos, 1994):

$$\Delta P = \frac{2\gamma_r}{r} + \rho gh + \Delta p_r \quad (10)$$

for MBP method and for DMBP method as:

$$\Delta P = \left(\frac{2\gamma_r}{r} + \rho gh + \Delta p_r \right) - \left(\frac{2\gamma_R}{R} + \rho gh + \Delta p_R \right) \quad (11)$$

Eq. (11) can be re-written as follows:

$$\Delta P = \left(\frac{2\gamma_r}{r} - \frac{2\gamma_R}{R} \right) + \left(\Delta p_r - \Delta p_R \right) \quad (12)$$

Hydrodynamic effects result from aerodynamic resistance of the capillary to the flowing gas and pressure changes during bubble formation as well as meniscus displacement (radius changes from the original value at time zero up to the moment when a hemispherical bubble is formed at the capillary tip) and hydrodynamic resistance of liquid against moving bubble. By using Stokes law for viscous resistance of the liquid it was found that the difference between apparent (measured) (γ_{app}) and true (γ_r) values of surface tension of the sample can be evaluated from the following Equation (Fainerman, Miller and Joos, 1994; Mobius and Miller, 1998):

$$\Delta\gamma = \gamma_{app} - \gamma_r = \frac{3\mu r}{2\tau} \quad (13)$$

where: “ μ ” is viscosity of liquid and “ τ ” is a bubble surface age. The value of “ $\Delta\gamma$ ” increases with increasing viscosity, increasing capillary radius and decreasing surface age. For liquids of low viscosity e.g. water, and diluted aqueous solutions the correction usually does not exceed ± 0.5 mN/m.

For viscous liquids, the contribution of hydrodynamic effect (and the resulting error) may be very significant and vary from a few to a few hundred per cent depending on the liquid viscosity, capillary size and dynamics of the bubbling process conditions at the orifices. For non-Newtonian liquids, there is another complicating factor – uncertainty of the local viscosity value in the vicinity of the capillary tip (shear thinning and shear thickening liquids). In addition, this viscosity may vary during the measurement. This may be a source of significant error as well.

Experimental

Water/glycerol solutions have been used as a model system for studying the effect of viscosity on the dynamic surface tension of viscous liquids. The values of surface tension of water and glycerol and their solutions are well known. By using different water/glycerol ratios, a wide range of liquid viscosity could be covered. In this paper, the viscosity range from 1 mPas up to 760 mPas (at 22°C) has been covered.

The dynamic surface tension of solutions has been measured using a Sensadyne PC500L instrument, by Chem Dyne Research Corporation, USA. The measurements were performed using the following gas flow rates at small orifice: 1, 2, 3, 4, 5 and 7 bubbles per second in pure water. Large orifice bubble rate was set constant at about 2 bubbles per second in pure water. Three different types of probes were used made of glass, stainless steel and Teflon. The capillary sets used in experiments had the following sizes: large probe diameter – 4 mm; small probe diameters - 0.5mm and 1.0 mm.

Results and Discussion

The values of apparent (measured) dynamic surface tension of water/glycerol solutions as a function of solution viscosity for different dynamic conditions are plotted in Fig. 3 and 4 for 0.5 mm and 1.0 mm glass capillaries, respectively. The real values of the DST are also presented for comparison (dotted line). As seen, the error in the determination of dynamic surface tension due to viscous effect is very significant when the viscosity of the solution is greater than 10 mPas. This error can be neglected for all dynamic conditions studied with

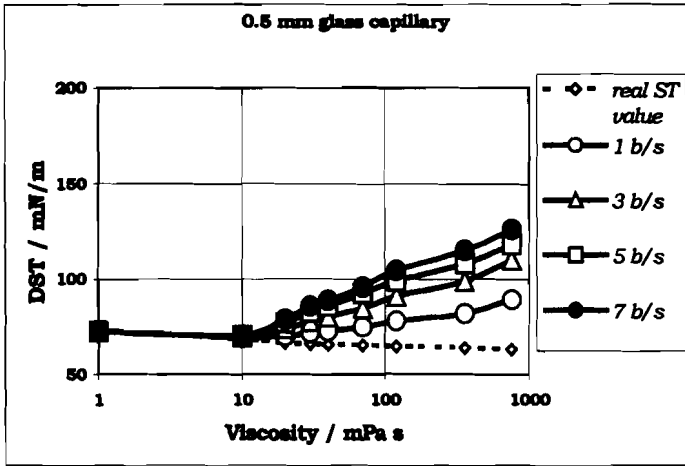


Fig. 3. The apparent dynamic surface tension as a function of liquid viscosity for different gas flow rates (expressed as a number of bubbles per second generated in pure water).

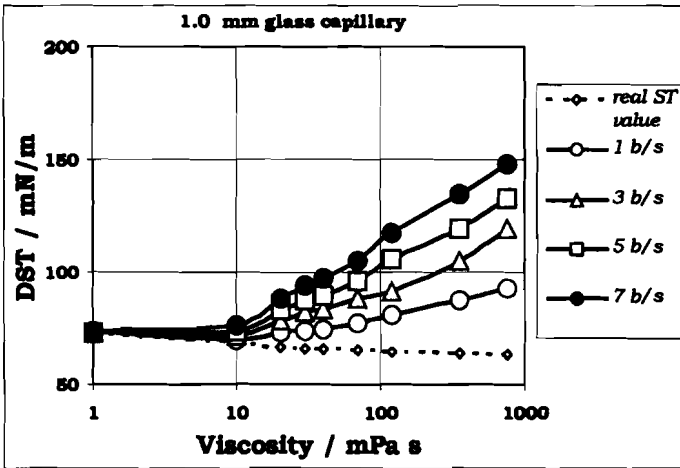


Fig. 4. The apparent dynamic surface tension as a function of liquid viscosity for different gas flow rates (expressed as a number of bubbles per second generated in pure water).

a 0.5 mm glass probe when solution viscosity is lower than 10 mPas. For higher solution viscosity, the deviations of apparent DST from the real value increase with increasing viscosity and bubble (lower surface age). For the gas flow equivalent to 7 bubbles/s in pure water and viscosity of 760 mPas, the error exceeds 100%. Similar behavior was observed for 1.0 mm glass capillary. For this system, measurable deviations were observed for viscosities lower than 10 mPas. The error for the most viscous sample studied at 7 bubbles/s exceeded 150 %.

The dynamic surface tension vs. solution viscosity plots for different dynamic conditions for 0.5 mm and 1.0 mm stainless steel capillaries are presented in Figs 5 and 6, respectively. As observed, the deviations from the real value of DST (dashed line) are very large and they are observed for viscosities higher than 1 mPas. For the solution of viscosity 760 mPas and bubble rate 7 bubbles per second in pure water, this error exceeds 200 % for 1.0 mm capillary. Such large deviations observed for glass and stainless steel capillaries can be due to the surface properties of these materials as well as surface roughness of stainless steel capillaries. The error increased with increasing viscosity and increasing capillary diameter.

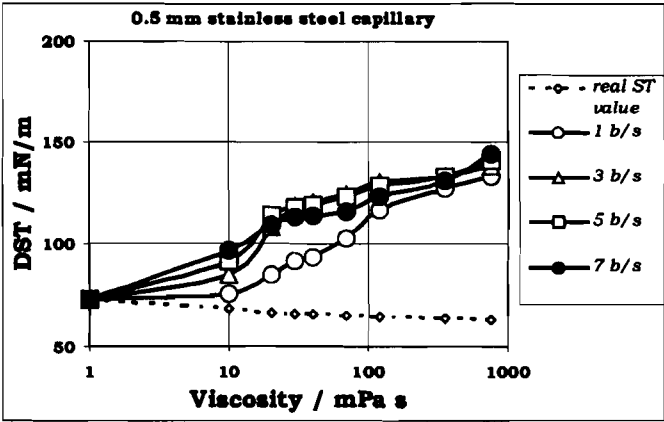


Fig. 5. The apparent dynamic surface tension as a function of liquid viscosity for different gas flow rates (expressed as a number of bubbles per second generated in pure water).

The plots of apparent DST vs. viscosity for different bubble rates for 0.5 mm and 1.0 mm Teflon capillaries are presented in Figs 7 and 8, respectively. As seen, the deviations of the DST from the real value for this system are much smaller than those observed for glass and stainless steel. By using a 0.5 mm

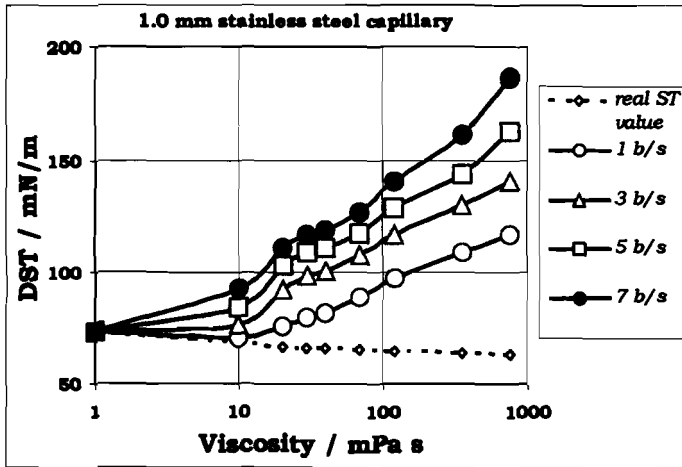


Fig. 6. The apparent dynamic surface tension as a function of liquid viscosity for different gas flow rates (expressed as a number of bubbles per second generated in pure water).

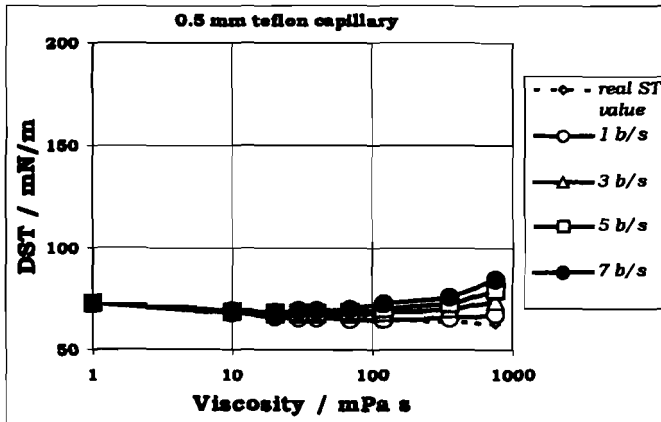


Fig. 7. The apparent dynamic surface tension as a function of liquid viscosity for different gas flow rates (expressed as a number of bubbles per second generated in pure water).

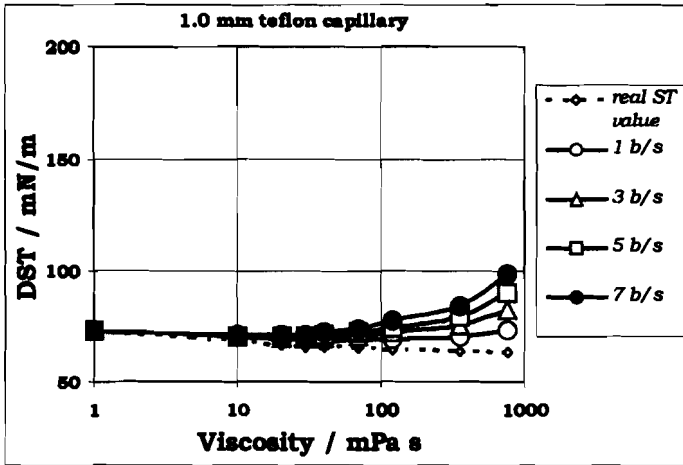


Fig. 8. The apparent dynamic surface tension as a function of liquid viscosity for different gas flow rates (expressed as a number of bubbles per second generated in pure water).

Teflon probe, the viscous effect error is negligible for viscosity lower than 20 mPas. Even for the most dynamic conditions studied (7 bubbles per second in pure water) and the highest viscosity (760 mPas) the error is about 25 % for 0.5 mm orifice and about 50 % for 1.0 mm capillary.

Comparison of the effect of material of the probe (glass, Teflon and stainless steel) on the viscous effect for two different capillary sizes – 0.5 mm and 1.0 – mm is presented in Figs 9 and 10, respectively. As seen, the error is the smallest for Teflon probes and the highest for stainless steel probes. This may result from different surface properties of the materials used and their effect on the mechanism of bubble growth and release from the capillary tip (Hofemeier, Yaminsky and Christenson, 1995; Mobius and Miller, 1998; Mysels, 1990). More evidence supporting this idea comes from the data presented in Figs 11 and 12. The values of the apparent DST vs. surface age of the bubble for different liquid viscosities are presented in Figs 11 and 12 for 0.5 mm glass capillary and 0.5 mm Teflon capillary, respectively. For the same bubble frequency (measured in pure water), the surface ages of bubbles generated with glass capillaries were longer than those for Teflon probes (for the liquid of the same viscosity). At the same bubble surface age the error in the DST value was much higher for glass capillary than for Teflon probe.

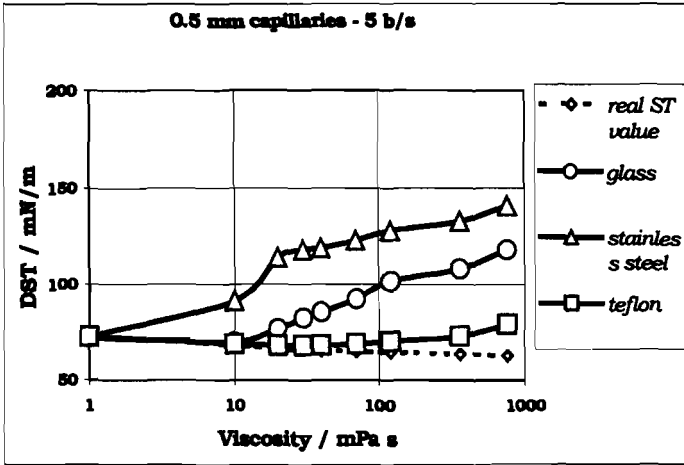


Fig. 9. The apparent dynamic surface tension as a function of liquid viscosity for constant bubble frequency (5 bubbles per second in pure water) and different probe materials.

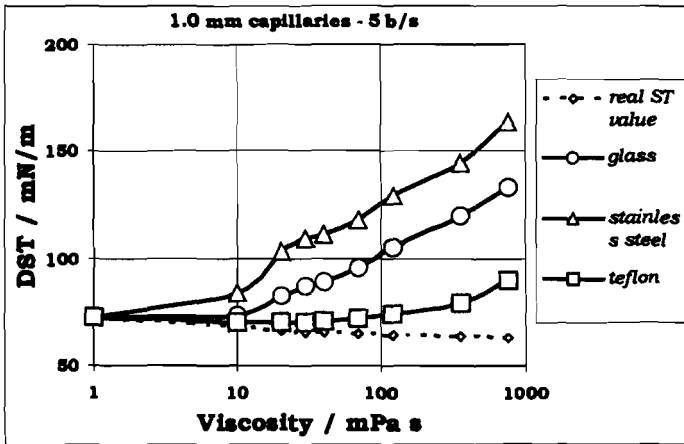


Fig. 10. The apparent dynamic surface tension as a function of liquid viscosity for constant bubble frequency (5 bubbles per second in pure water) and different probe materials.

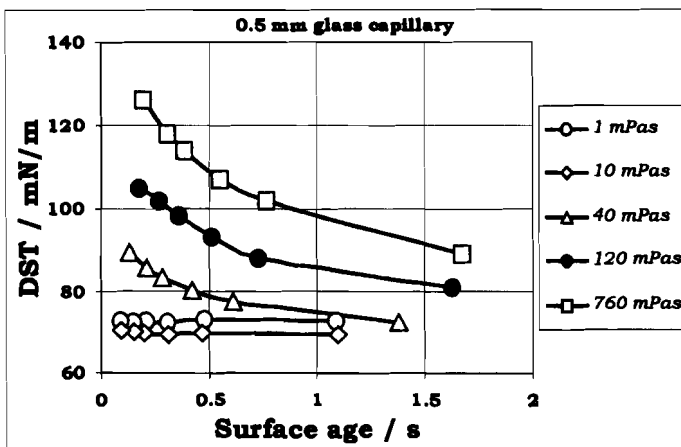


Fig. 11. The apparent dynamic surface tension as a function of bubble surface age for different viscosities of liquids.

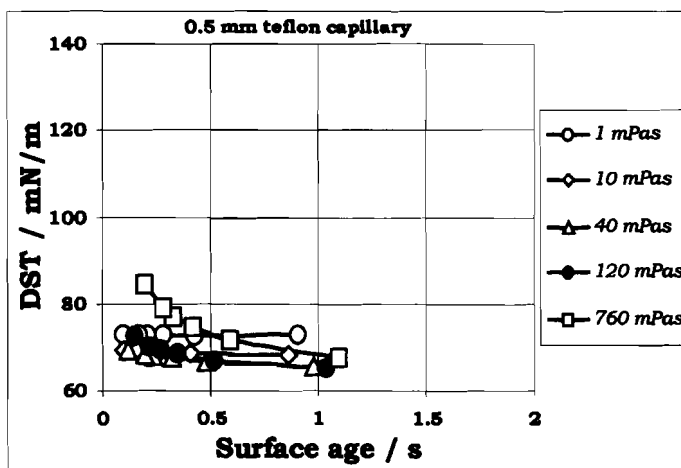


Fig. 12. The apparent dynamic surface tension as a function of bubble surface age for different viscosities of liquids.

The data on the viscous effect on the DST measurements presented in this paper clearly show that the error due to this effect can be very high and much larger than from any other sources discussed above. For a reasonable evaluation of the dynamic surface tension of the viscous liquid sample using the DMBP method,

this effect has to be taken into account. The simplest solution may be use of model viscous liquids. In a first step, one can measure the DST of the model liquid (e.g. water/glycerol solutions) under different dynamic conditions using a given set of probes and compare the apparent DST to the real DST values taken from the literature or measured under equilibrium conditions. It is noteworthy that for water/glycerol solutions, the values of the dynamic and equilibrium surface tensions are the same. Next, one can subtract the difference between apparent and real values of the DST for a model liquid (under the same dynamic conditions and for the same probes) from the apparent value of the DST of the unknown sample of the same viscosity as the model liquid. Better accuracy can be obtained when using Teflon probes. The example results obtained in this way for different viscous samples are presented in Table 1.

Table 1. Comparison of apparent (viscous effect) and corrected values of the dynamic surface tension for a few selected systems. Large orifice – Teflon 4 mm, small orifice – Teflon 0.5 mm, gas flow rate – 5 bubbles/s in pure water.

| Sample | Viscosity (mPas) | Apparent DST (mN/m) | Corrected DST (mN/m) |
|------------------|------------------|---------------------|----------------------|
| Polymer solution | 60 | 36 | 33 |
| Polymer solution | 280 | 38 | 32 |
| Water-based ink | 60 | 41 | 38 |
| Polymer solution | 180 | 42 | 37 |

Such an approach was used before for the MBP method (Fainerman, Makievski and Miller, 1993). Since very narrow capillaries are used in the MBP method the error due to viscous effect is much smaller than that observed for larger capillaries and the DMBP method. As mentioned above, all experimental errors will not cancel out in the DMBP method (two capillaries used).

The data presented in this paper are very preliminary and cannot be compared to any other data because such data are not available in the literature for the DMBP method. More work has to be done to further evaluate the effect of different parameters (surface properties of liquids, probe type and shape, etc) on the error due to viscous effect.

Recently, a new instrument for measuring dynamic surface tension of viscous liquids has been introduced and is available on the market – Sensadyne PC500LV, by Chem Dyne Research Corporation, USA. The manufacturer claims that this instrument is capable of accurate measurements of the DST of viscous liquids (automatic viscosity compensation) (Janule, 2001).

Summary/Conclusions

The effect of viscosity on the dynamic surface tension measurements of liquids has been studied using water/glycerol solutions as a model system. Based on the experimental results described in this paper, the following conclusions can be drawn:

1. The viscous effect can be a source of a very significant error in determination of the dynamic surface tension of viscous liquids using DMBP method.
2. The value of error for the systems studied ranged from 1 % to ~250 % depending on the experimental setup, dynamic conditions and viscosity.
3. The smallest errors (most accurate results) were obtained using Teflon probes.
4. To improve the accuracy of the DST measurements of viscous liquids, the correction factors obtained for the model liquid can be subtracted from the apparent DST value of a test sample (under the same dynamic, probe and viscosity conditions) and thus viscous effect error can be minimized.

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