# Effect of Polymer/Surfactant Interactions on Rheological Properties of Polymer/Surfactant Mixtures

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## Abstract

Surface tension and rheological properties are amongst the most critical factors driving spreading and leveling of inks and coatings. In water-based systems, surfactants are commonly used to control surface tension. Due to specific interactions with the polymers present in the inks, surfactants may have a significant impact on ink rheology. The effect of the interactions between polymers (SMA® 1000H; SMA® 1440H; Joncryl® 63 and Joncryl® 89) and acetylenic diol surfactants (Surfynol® 104H and Surfynol® 440) on rheological properties of water-based inks was studied using viscosity and flow measurements. Two samples of water-based pigmented inks having good and bad printability were used. Both inks contained three different levels of solids: press ready ink; virgin ink and "waterless" ink (no free water added during formulation). All inks had identical composition except for the nature of the surfactant used. Effect of interactions between polymer and surfactant on the rheology of polymer-surfactant mixture seems to be dependent on chemistry of both surfactant and polymer. For a given polymer ethoxylated surfactant affected viscosity more than the non-ethoxylated one and for a given surfactant interactions with hydrophobic polymers affected viscosity more than interactions with hydrophilic polymer. Viscosity of polymer solution-surfactant mixtures could be higher, unchanged or lower than the viscosity of the virgin polymer solution. For polymer dispersion viscosity of polymer-surfactant mixture was never lower than the viscosity of the original varnish. Surfactants may affect very strongly ink flow properties at low shear rates and thus may be one of dominating factors controlling printability.

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## Introduction

One of the factors responsible for good printability of liquid inks is their low surface tension. Low surface tension is indispensable for good spreading of the ink on the substrate surface. To achieve good spreading surface tension of liquid ink and interfacial tension between the ink and the substrate should be as low as possible while surface tension of the substrate should be as high as possible (Rosen, 1978; Ross and Morrison, 1988)

Solvent borne inks have inherently low surface tension due to solvent used e.g. alcohols, toluene, etc. Though surface tension of water is high (~73 dyne/cm) the surface tension of water based inks can be lowered by the addition of the appropriate surfactants. The most popular are non-ionic surfactants due to their high effectiveness (low dynamic surface tension - DST - and foaminess).

Aqueous solutions of surfactants achieve the minimum value of surface tension at the so-called critical micelle concentration (cmc). At the cmc concentration of free surfactant molecules in the solution reaches a maximum. Above the cmc process of association of surfactant molecules and formation of micelles is more energetically favorable. Some surfactants do not form micelles due to their molecular structure and for such surfactants their solubility in water determines the concentration of free surfactant molecules in the solution. The values of the cmc (or solubility) vary dependent on the character of surfactant (anionic, cationic, non-ionic) and chemical structure (Rosen, 1978) but they are roughly in the range 0.01 - 0.2 %.

In polymer solutions the minimum value of the dynamic surface tension is achieved at considerably higher concentration of surfactant. This is because of interactions between surfactant and polymer molecules. Extent of such interactions depends on chemistry of both polymers and surfactants (Goddard, 1993a; 1993b; 1993c; 1994; Kevelam et. al., 1996; Nahringbauer, 1996; Saito, 1987). In the real systems to achieve required value of the DST of water based ink surfactant doses can be as high as one percent or higher.

Interactions between polymer and surfactant affect surface tension of water based ink and may also affect its rheological properties. It is known from the literature that viscosity of polymer solution may increase significantly as a result of such interactions (Goddard, 1994; Lindman and Thalberg, 1993; Saito, 1987). Most of literature data refer, however, to the diluted polymer solutions. In liquid inks concentration of polymers is high and other ingredients such as pigments, fillers, surfactants and other additives are present as well. Literature data on polymer/surfactant interaction in such systems are rather rare (Krishnan and Sprycha, 1999; Sprycha and Krishnan, 1998).

Both, surface chemistry and rheology are very important in understanding very complex phenomena occurring during printing (Gilleo, 1991; Tadros, 1996). For example, viscosity of ink during printing may change depending on the forces exerted on the ink. This is because the printing inks are not ideal Newtonian liquids. For such systems the shear force directly affects the viscosity of printing inks. In fact during printing one is dealing with a viscosity curve rather than a fixed viscosity value.

Most of printing inks undergo "shear thinning" phenomenon on the printing press. As shear rate increases the ink viscosity drops sometimes dramatically. Shear forces of different magnitude act on the ink during wiping process (doctor blade), in the printing nip (filament formation), and during spreading and leveling. Important factor is also time dependency. For some inks their viscosity is dependent on the value of the shear forces applied and on the length of time. If shearing is stopped the ink will return to its initial viscosity. The time in which the ink achieves its original viscosity can very from a fraction of a second to hours.

One of the important factors determining ink flow is yield value. Ink rheology should be considered as a dynamic variable and studied how it changes throughout the printing process (flexography, gravure, and lithography). Yield point controls, for example, ink "bleeding". Low yield value will provide excellent ink flow but sometimes "bleeding" may be excessive. On the other hand high yield value will prevent ink flow. Yield value depends on ink composition - polymers, pigments, fillers, and additives. Leveling of inks and coatings is to a large extent dependent on yield value. During leveling shear stress due to surface tension forces is relatively low. If the yield value is higher than such stress flow will not take place and liquid will behave like solid. In many practical situation poor leveling is due to too high yield value. This value may be an intrinsic property of the system and if so only reformulation can change ink or coating behavior. High yield value may be unwanted phenomenon preventing good leveling of the ink in solid areas (continuous ink film is formed) but it may be desired while printing mid and light tones (low dot gain). In practice, both yield value and surface tension are important and have to be balanced to obtain expected ink flow and spreading.

Studying of flow properties at low shear rate and evaluation of yield value for low viscosity printing inks e.g. gravure or flexographic inks, is not an easy task because relatively weak forces have to be measured. On the other hand one has to remember that viscosity of such inks may vary dramatically upon ink setting on paper. In the very first moment of ink transfer in the printing nip two processes responsible for solvent removal start simultaneously - evaporation and absorption into the paper. These processes run at different rates but between the

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nip and the dryer sorption usually prevails. Removal of solvent causes locally significant change in the concentration of polymer, surfactant and pigment. Such changes may cause dramatic changes of rheological properties of the ink and thus influence ink spreading, flow and leveling.

To better understand the effect of polymer/surfactant interactions on the rheological properties of printing inks viscosity and flow measurements were performed in the model systems. Polymer/surfactant mixtures of different polymer:surfactant ratios and concentrations were used. Model system consisted of relatively well-defined polymers (styrenated maleic anhydride resins) and acetylenic diol surfactants. In addition, similar measurements were performed using styreanted acrylic solutions and dispersion and two water based inks containing different levels of solids: press ready inks; virgin inks and inks formulated without water addition.

## Experimental

### Materials

Aqueous solutions of two different styrenated maleic anhydride resins (SMA® 1000H and SMA® 1440H) were obtained from Elf Atochem, Inc., USA. SMA® 1000H is ammonia neutralized solution of SMA® 1000 resin in which styrene:maleic anhydride ratio is 1:1. SMA® 1440 H is ammonia-neutralized solution of the half ester of SMA® 1000 and butoxyethanol. In addition, two acrylic polymers obtained from SC Johnson, USA were used: Joncryl® 63 - ammonia neutralized solution of low molecular weight (~12,000) Joncryl® 67 resin and Joncryl® 89 - styrenated acrylic dispersion (molecular weight >200,000).

Two acetylenic diol surfactants obtained from Air Products and Chemicals, Inc., Allentown, Pennsylvania, USA were used in experiments. Surfynol® 104H is non-ethoxylated version of (2,4,7,9-tetramethyl - 5 - decyne - 4,7 - diol) and Surfynol® 440 contained ~40% by weight of ethylene oxide (~3.5 mole).

### Methods

Polymer solutions of four different concentrations (1%, 3%, 10% and 20% w/w solids) were prepared using de-ionized water. Viscosity of polymer/surfactant mixtures was measured using efflux cups. 300g of polymer solution of a given concentration was agitated in a glass jar with a stirrer and surfactant was added to the system in small portions. After each surfactant addition the solution was stirred for 20 minutes (Surfynol® 104H) or 10 minutes (Surfynol® 440) until the "equilibrium" was reached. The time to reach equilibrium was determined in the

separate experiments. Then mixer was stopped and viscosity was measured using efflux cups. Surfactant was added stepwise until its excess in the system was observed - solution cloudiness.

Flow measurements were performed using stress-controlled rheometer AR-1000N, TA Instruments, USA on the samples of "Good" and "Bad" inks and on original varnishes of different level of solids (as obtained from the supplier): 36% - SMA® 1000H; 33% - SMA® 1440H; 31% - Joncryl® 63 and 49% - Joncryl® 89. The amount of surfactant added to the polymer solution was equal to 5%. The measurements were performed at constant temperature 25°C.

## Results and Discussion

Surfynol® surfactants based on acetylenic diol chemistry represent a unique category of surfactants, which provide low dynamic surface tension and a very good defoaming, and surface wetting properties ( Dougherty, 1989; Medina and Sutovich, 1994). Though these surfactants are widely used in the ink and coating industry there is a little information available on the interaction of acetylenic diols with other ink/coating ingredients such as polymers, pigments, etc. It was found previously (Krishnan and Sprycha, 1999; Sprycha and Krishnan, 1998) using DST measurements that acetylenic diol surfactants can interact very strongly with polymers forming less surface-active complexes than the surfactant. Extent of interactions depended on chemistry of polymers and surfactants. Interactions increased with increasing hydrophobicity of polymers and degree of ethoxylation of surfactant. Both, hydrophobic and hydrogen bond interactions seemed to be involved in the interactions between polymer and surfactant molecules. It was also observed that at higher polymer and surfactant concentrations viscosity of such mixtures increased. At that time, however, no quantitative viscosity or flow measurements were performed.

In this paper the effect of polymer/surfactant interactions on rheology of polymer/surfactant mixtures was evaluated by different methods. For quick evaluation of viscosity efflux cups were used. Though efflux cups give rather limited information about the system they are commonly used in the industry.

The viscosity of SMA® 1000H polymer solutions of different concentrations as a function of the amount of surfactant added to the system is presented in Figs 1 and 2 for Surfynol® 104H and Surfynol® 440, respectively. As seen Surfynol® 104H had almost no effect on polymer solution viscosity in the entire polymer and surfactant concentration range studied. On the other hand substantial increase of solution viscosity was observed for Surfynol® 440 surfactant and 20% solution of SMA® 1000H. Before the excess of free surfactant in the system was observed after addition ~18 g of surfactant viscosity reached
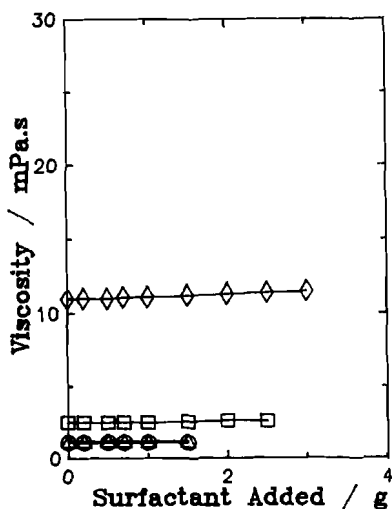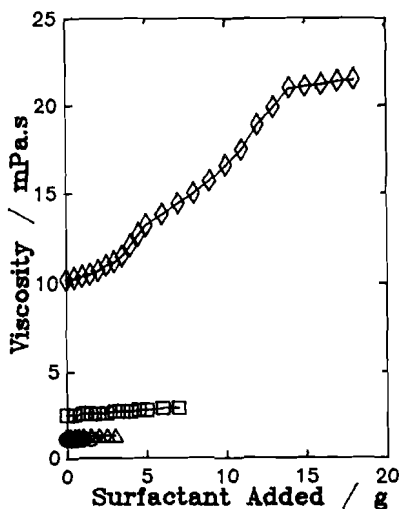
Fig. 1



Fig. 2

Fig. 1    Viscosity of polymer solution vs. surfactant dose for SMA®
         1000H/Surfynol® 104H system. Polymer concentrations:  O, 1%;  Δ ,
         3%;  □ , 10%; ◊ , 20 %.

Fig. 2    Viscosity of polymer solution vs. surfactant dose for SMA®
         1000H/Surfynol® 440 system. Polymer concentrations:  O, 1%;  Δ ,
         3%;  □ , 10%; ◊ , 20 %.

"plateau". This may indicate that under a given experimental conditions all
available for adsorption/interaction sites on the polymer chains were blocked by
the surfactant molecules and no more complexes could be formed (Goddard,
1994; Krishnan and Sprycha, 1999). The results presented in Figs 1 and 2
clearly show that the higher surfactant ethoxylation degree the stronger effect of
polymer/surfactant interactions on polymer solution viscosity.

For more hydrophobic polymer, SMA® 1440H, interaction between polymer
and non-ethoxylated surfactant molecules caused significant increase of 20%
solution of polymer - Fig. 3. At the same time total "consumption" of surfactant
was 18 g compare to 3 g for SMA® 1000H polymer. When ethoxylated
surfactant was added to the SMA® 1440H polymer solution strong effect of
polymer/surfactant interactions on polymer solution viscosity was observed
already for 10% solution of polymer - Fig, 4. Amount of surfactant "consumed"
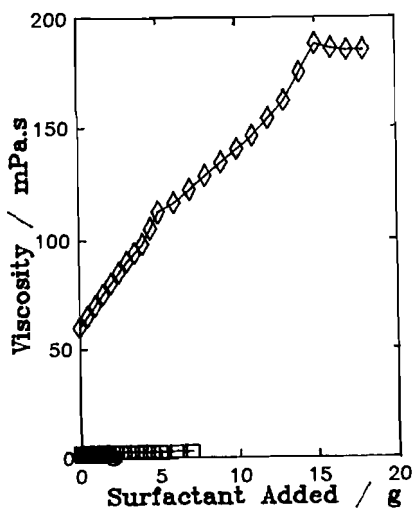
Fig. 3

Fig. 4

Fig. 3    Viscosity of polymer solution vs. surfactant dose for SMA®
1440H/Surfynol® 104H system. Polymer concentrations:  O, 1%;  Δ ,
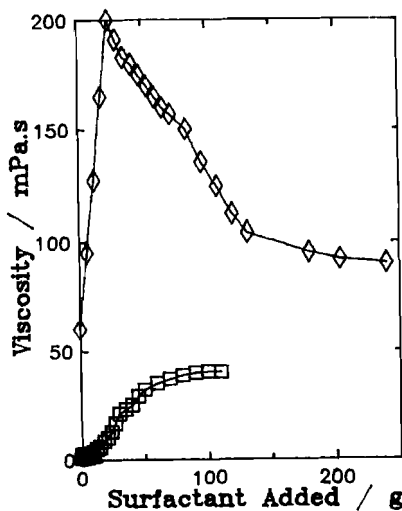3%;  □ , 10%; ◊ , 20 %.

Fig. 4    Viscosity of polymer solution vs. surfactant dose for SMA®
1440H/Surfynol® 440 system. Polymer concentrations:  O, 1%;  Δ ,
3%;  □ , 10%; ◊ , 20 %.

by polymer (complex formation) was equal to 120 g. Surprising results were
obtained for 20% solution of SMA® 1440H. At first, very rapid increase in
polymer solution viscosity was observed and maximum on the curve was
reached after addition of 25 g of Surfynol® 440 surfactant. For higher doses of
surfactant polymer solution viscosity decreased quite rapidly. Such behavior is
indicative of very dramatic changes in the system. Viscosity of polymer solution
depends on conformation of polymer chains and their interactions with other
solution ingredients. In polymer/surfactant mixtures viscosity depends on
interactions between solution species including surfactant/polymer complexes.
Under certain conditions polymer chains (or polymer-surfactant complexes)
may show tendency to coiling and thus viscosity of such system should be lower
than that for original solution. Though aggregates formed may affect
significantly viscosity, they can be invisible by naked eye due to very small size
of such "species" and lack of light scattering in the visible range - no turbidity.
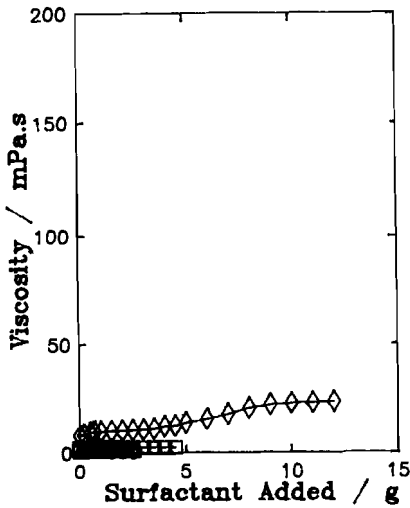
Fig. 5

Fig. 6

Fig. 5    Viscosity of polymer solution vs. surfactant dose for Joncryl®
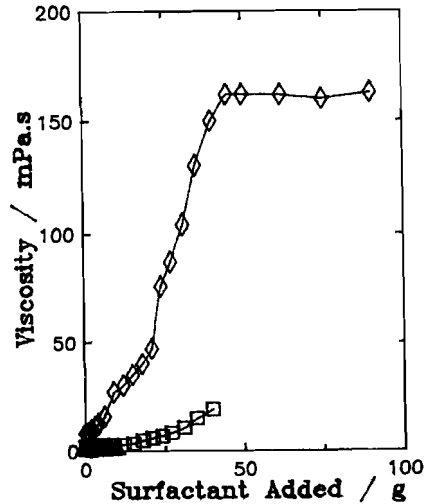63/Surfynol® 104H system. Polymer concentrations:  O, 1%;  △ , 3%;
□ , 10%; ◊ , 20 %.

Fig. 6    Viscosity of polymer solution vs. surfactant dose for Joncryl® 63
/Surfynol® 440 system. Polymer concentrations:  O, 1%;
△ , 3%;  □ , 10%; ◊ , 20 %.

Such phenomena may be responsible for the shape of the curve for 20% solution
of SMA® 1440H as observed in Fig. 4.

For Joncryl® 63 solutions minimal effect of polymer/surfactant interactions on
polymer solution viscosity was observed for Surfynol® 104H surfactant and
20% polymer solution - Fig. 5. However, substantial increase in polymer
solution viscosity was observed for Joncryl® 63/Surfynol® 440 system - Fig. 6.
For the latter system viscosity of 20% polymer solution increased from initial
value of 10 mPas to 165 mPas ("plateau" on the curve).

Due to lower viscosity of polymer dispersions (at the same level of solids) range
of concentration for Joncryl® 89 was expanded up to 30% solids. However, even
at this concentration no effect of polymer/surfactant interactions on dispersion
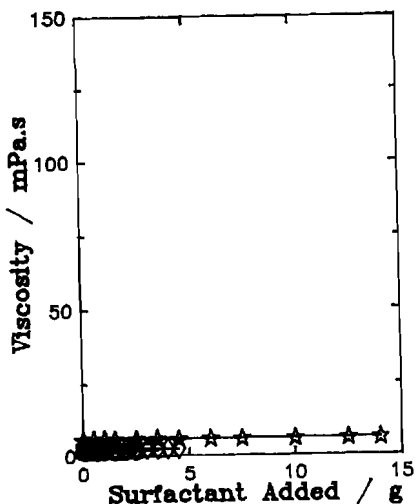viscosity was observed for Surfynol® 104H surfactant - Fig. 7. For ethoxylated

Fig. 7

Fig. 8

Fig. 7     Viscosity of polymer solution vs. surfactant dose for Joncryl® 89
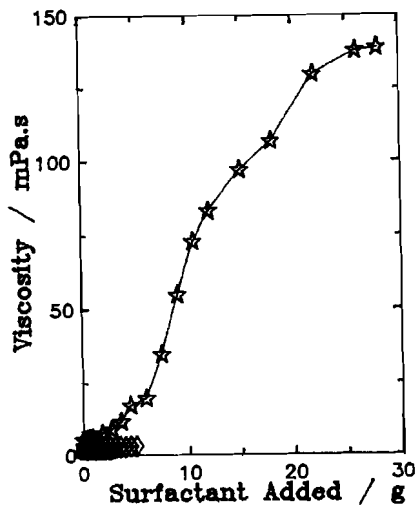/Surfynol® 104H system. Polymer concentrations: O, 1%; Δ , 3%; □
, 10%; ◊ , 20 %; ☆ ,30%.

Fig. 8     Viscosity of polymer solution vs. surfactant dose for Joncryl® 89
/Surfynol® 440 system. Polymer concentrations: O, 1%; Δ , 3%; □ ,
10%; ◊ , 20 %; ☆ , 30%.

surfactant (Surfynol® 440) and 30% dispersion of Joncryl® 89 significant
increase of polymer dispersion viscosity was noticed with increasing doses of
surfactant - Fig. 8.

The results presented in Figs 1-8 show that interactions between polymer and
surfactant molecules at higher polymer concentrations could affect very
significantly the viscosity of polymer solution (dispersion). To further elucidate
this problem flow measurements were performed for more concentrated (> 30%
solids) polymer solutions (dispersions). The results obtained for SMA® 1000H
polymer solution are presented in Fig. 9. As seen the curves for virgin SMA®
1000H solution and the same solution containing 5% of Surfynol® 104H
surfactant run parallel to each other and the only effect observed was lower
viscosity of polymer-surfactant mixture compared to polymer solution itself.
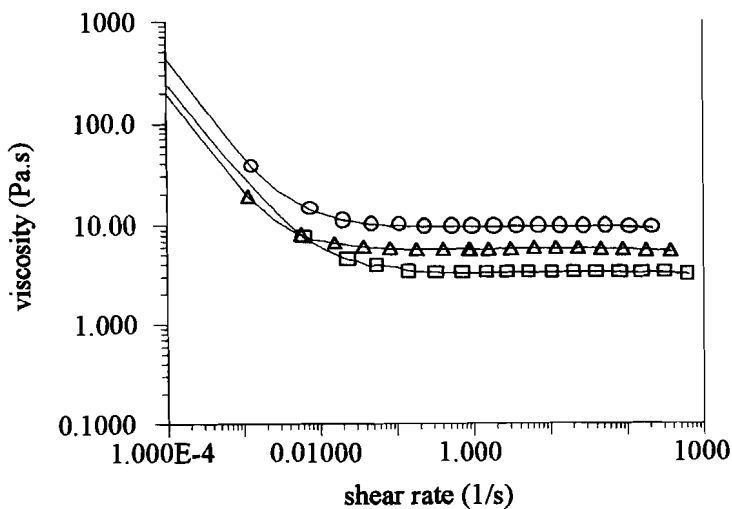Similar lowering of viscosity was observed for 20% solution of SMA®

Fig. 9    Viscosity of SMA® 1000H virgin polymer solution vs. shear rate: O - no surfactant added; Δ - 5% Surfynol® 104H added; □ - 5% of Surfynol® 440 added.
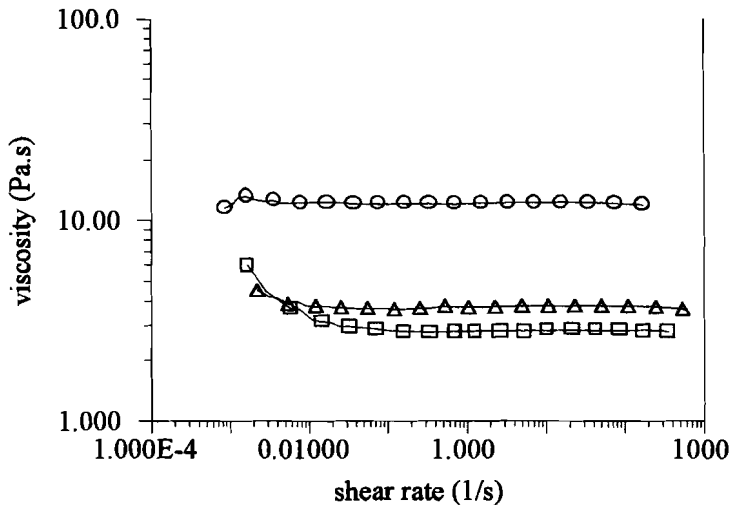


Fig. 10   Viscosity of SMA® 1440H virgin polymer solution vs. shear rate: O - no surfactant added; Δ - 5% Surfynol® 104H added; □ - 5% of Surfynol® 440 added.

1440H/Surfynol® 440 system for higher surfactant doses - see Fig. 4. Addition of 5% of Surfynol® 440 to SMA® 1000H solution caused even bigger drop in viscosity. For this system, however, curve does not run parallel to the other curves and the rate of viscosity increase, for low shear rates (below 1.0 s⁻¹), was higher than that for other systems.

Different relationships between viscosity and shear rate were observed for SMA® 1440/Surfynol® 104H(440) systems - Fig. 10. In the entire range of shear rates measured all systems except SMA® 1440H/Surfynol® 440 mixture showed quasi-Newtonian behavior. In the latter system increase in viscosity was observed when the shear rate dropped below 0.1 s⁻¹. Similar to SMA® 1000H/surfactant systems addition of surfactant caused substantial drop of solution viscosity.

Virgin Joncryl® 63 polymer solution showed some shear thinning for low shear rates (<0.1 s⁻¹) - Fig. 11. After addition of Surfynol® 104H surfactant the system showed quasi-Newtonian behavior. On the other hand, addition of Surfynol® 440 (ethoxylated surfactant) resulted in more shear thinning than that observed for virgin Joncryl® 63 solution. It is noteworthy that for shear rates in the range from 0.1 s⁻¹ up to 500 s⁻¹ all the curves almost coincided i.e. very slight
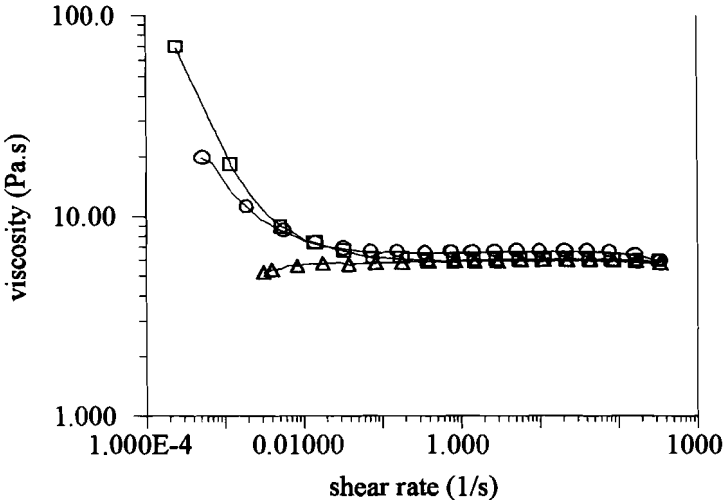


Fig. 11    Viscosity of Joncryl® 63 virgin polymer solution vs. shear rate: O - no surfactant added; Δ - 5% Surfynol® 104H added; □ - 5% of Surfynol® 440 added.
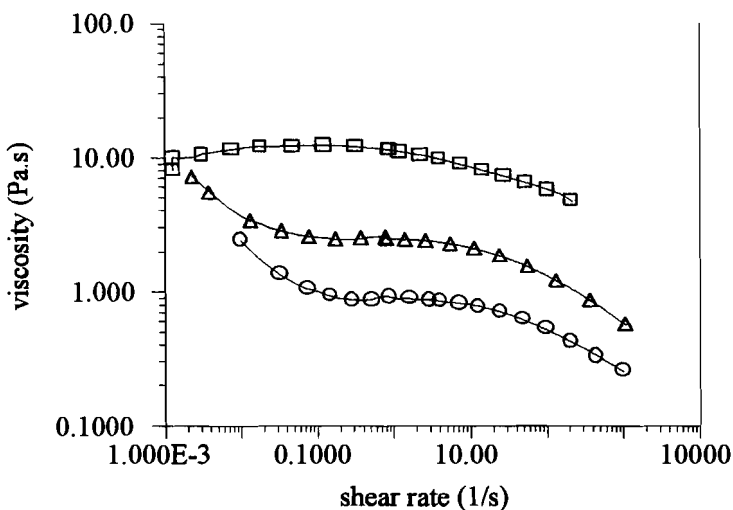
Fig. 12    Viscosity of Joncryl® 89 virgin polymer dispersion vs. shear rate: O - no surfactant added; Δ - 5% Surfynol® 104H added; □ - 5% of Surfynol® 440 added.

difference in viscosity was observed between different systems. Such behavior is significantly different from that observed for styrenated maleic anhydride polymer solutions/Surfynol® 104H(440) systems where addition of surfactant caused significant drop in solution viscosity.

Joncryl® 89 dispersion showed shear thinning behavior with primary "plateau" in the shear range 0.1 - 10 s$^{-1}$ - Fig. 12. Addition of Surfynol® 104H surfactant caused viscosity increase but shape of the curve remained almost unchanged. Mixture of Joncryl® 89 and Surfynol® 440 behaved differently than two other systems. Generally viscosity of that polymer-surfactant mixture was significantly higher than that for virgin Joncryl® 89  dispersion and Joncryl® 89/Surfynol® 104H systems. In addition, no shear thinning was observed for very low shear rates. In the range 10$^{-3}$ - 1.0 s$^{-1}$ the mixture showed close to Newtonian behavior.

Flow curves for "Good" and "Bad" inks are presented in Figs 13 and 14, respectively. As seen for cut (press ready) and virgin inks there were almost no differences between  "Good" and "Bad" samples. Both inks showed close to Newtonian behavior. Due to low ink viscosity measurements could not be performed at low shear rates below 1 s$^{-1}$. Such measurements were, however, performed for "waterless" inks of much higher viscosity. As seen in Figs 13
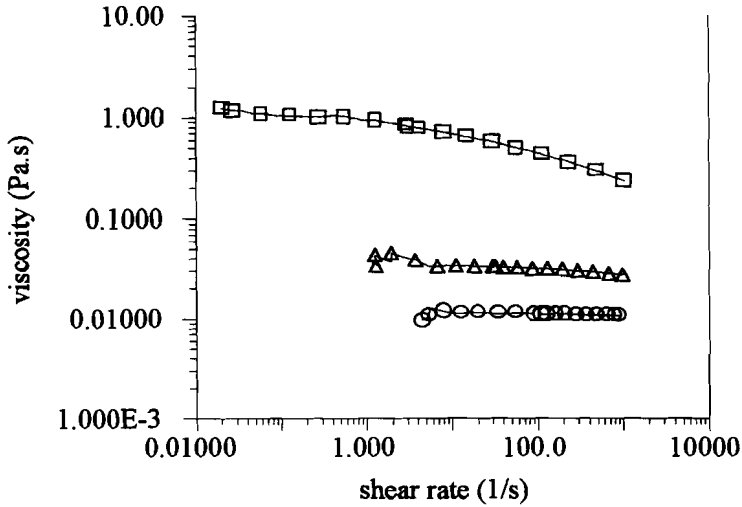
Fig. 13    Viscosity of "Good" ink vs. shear rate: O - press ready ink; Δ - virgin ink; □ - "waterless" ink (no water added during formulation).
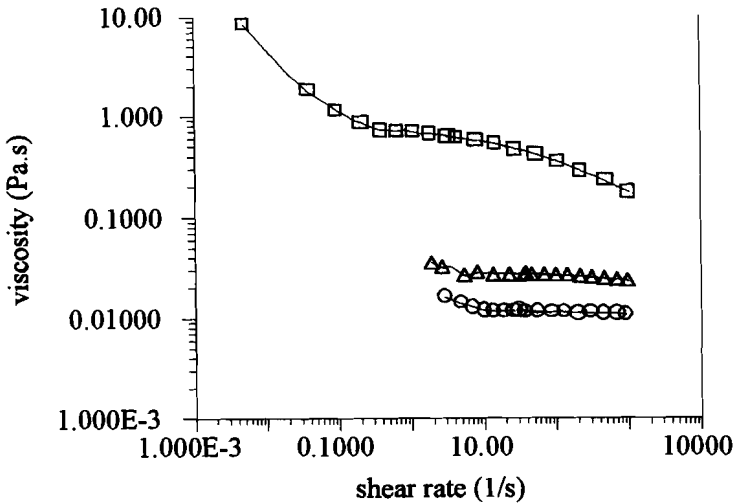


Fig. 14    Viscosity of "Bad" ink vs. shear rate: O - press ready ink; Δ - virgin ink; □ - "waterless" ink (no water added during formulation).

and 14 "Good" ink showed close to Newtonian behavior in the shear range $10^{-3}$-$1.0\ s^{-1}$ while viscosity of "Bad" ink increased significantly with decreasing shear rate. Thus, one may expect that "Good" ink will flow nicely during leveling while "Bad" ink (more structured one) will show poor leveling. It is noteworthy

that "Good" and "Bad" inks had identical compositions and level of surfactants and the only difference between them was different surfactant chemistry.

Forces acting between pigment and polymer particles control the rheological behavior of dispersions (such as a printing ink). These forces may have different origin - van der Waals, electrostatic, steric hindrance, hydrogen bond bridging - and be responsible for flocculation and coagulation. Depending on the extent and type of the interactions between different ink components a given ink may be stable or unstable dispersion (Tadros, 1996), be more or less structured and thus exhibiting different flow profiles (viscosity vs. shear rate plots). Shear rate during printing may vary and be as high as $10^4$-$10^6$ s$^{-1}$ (depending on the printing process) and as low as $10^{-4}$ -$1.0$ s$^{-1}$ during ink leveling and film formation.

Results presented in this paper showed that the interactions between polymers and surfactants and their effect on rheological properties of polymer-surfactant mixtures are indeed very complex issues. Viscosity of polymer solution-surfactant mixture may be higher, unchanged or lower than the viscosity of the original polymer solution. In addition, viscosity of such mixtures may or may not increase at very low shear rates (below 1 s$^{-1}$). Non-ethoxylated surfactant (Surfynol® 104H) had less effect on viscosity of polymer-surfactant mixtures than the ethoxylated surfactant Surfynol® 440. For a given surfactant its interactions with more hydrophobic polymer affected viscosity more than the interactions with hydrophilic polymer. For polymer dispersions (Joncryl® 89) under all experimental conditions interactions with both surfactants never caused viscosity decrease. Due to complexity of the subject and lack of literature data for such systems as described in this paper more experimental investigation is necessary before more general conclusions can be drawn.

### Conclusions

Effect of polymer/surfactant interactions on rheological properties of polymer-surfactant mixtures has been studied using viscosity and flow measurements. Three polymer solutions (SMA® 1000H; SMA® 1440H and Joncryl® 63) and one polymer dispersion (Joncryl® 89) and two surfactants (Surfynol® 104H and Surfynol® 440) were selected as a model systems for investigation, The findings in the present paper may be summarized as follow:

1.  Effect of interactions between polymer and surfactant on the viscosity of polymer-surfactant mixture depended on chemistry of both surfactant and polymer.

a).     for a given polymer ethoxylated surfactant affected viscosity more than the non-ethoxylated one.

b).     for a given surfactant interactions with hydrophobic polymers affected viscosity more than interactions with hydrophilic polymer.

2.     For polymer solutions viscosity of polymer-surfactant mixtures can be higher, unchanged or lower than the viscosity of the virgin polymer solution.

3.     For polymer dispersion viscosity of polymer-surfactant mixture was never lower than the viscosity of the original varnish.

4.     Surfactants may affect very strongly ink flow properties at low shear rates and thus may be one of dominating factors controlling printability.

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