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**New water-based copolymer nano- particles and their use
as eco-friendly binders for industry of flexographic ink, part
I**

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: New water-based copolymer nano- particles and their use as eco-friendly binders for industry of flexographic ink, part I: [Acrylate co-polymers, nano-emulsion, water-based flexographic inks, Binders, industry of inks](#)

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paper._RESEARCH_LIMITATIONS/IMPLICATIONS__(LIMIT_100_WORDS) :No data

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New water-based copolymer nano- particles and their use as eco-friendly binders for industry of flexographic ink, part I

Abstract

Purpose - The purpose of this paper is to prepare, characterise and evaluate nano-emulsions of co-polymers of various compositions as eco-friendly binders for flexographic ink industry.

Design/methodology/approach - Various nano emulsions of copolymers were prepared using St, BuAc, AA and AAm monomers by means of a conventional seeded emulsion polymerisation technique, using $K_2S_2O_8$ as the initiator. The characterisation of the prepared emulsions was performed using FT-IR, TGA, GPC, and TEM. A selection of co-polymers were formulated with pigments and additional ingredients, as water-based flexographic inks. The inks were characterised for their viscosity, pH, degree of dispersion, water resistance, and colour density.

Findings - It was found that the low viscosity of the prepared copolymers may reduce the film thickness of the flexographic inks and may also decrease the spreading of the ink on the surface. As a result, stable modified poly acrylate-based latex with improved physico-mechanical properties were obtained. The prepared latexes showed improved and enhanced water resistance, the gloss values, the print density which ranged from 2.06 to 2.51 and the maximum gloss values (39 and 48) were also obtained. Also, these binders provide excellent adhesion properties for both the pigment particles and the base paper.

Research limitations/implications - The study focuses on the preparation of new water-based copolymer nano-particles and their use as eco-friendly binders for flexographic ink industry.

Social implications - The ink formulations developed could find use in industrial scale printing.

Originality/value - Eco-friendly environment ink formulations for printing on paper substrates are novel.

Keywords: Acrylate co-polymers, nano-emulsion, water-based flexographic inks, binders, industry of inks.

Introduction

The ink industry is one of the world's growth markets, due to the increasing demand

1
2
3 for printing inks for packaging applications [Izdebska, 2016]. Inks are generally made
4 of four major components; binders, pigments, solvent (or dispersion media) and a
5 variety of property enhancement additives. Based on their compositions, inks may be
6 classified into two categories namely, solvent-based inks (formulated using volatile
7 organic compounds as solvents) and water-based inks (formulated using water as the
8 solvent). The presence of volatile organic compounds in solvent-based inks makes their
9 use less environmentally friendly than water-based inks. This has led to the increase in
10 demand for water-based inks [Zolek, 2015]. Water-based printing inks have been
11 successfully used in different printing applications on various surfaces or substrates
12 [Izdebska, 2016]. Previous studies have shown that the type and amount of different
13 components used in the formulation of printing inks, such as the polymer binders, have
14 effects on the properties of the ink [Pekarovicova, 2016]. There are also studies that
15 show that different additives improve the print properties of water-based ink
16 formulations [Zolek, 2015]. The three additives generally used in water-based ink
17 formulations are: (1) surfactants, (2) defoamers and (3) waxes. Surfactants are added to
18 reduce the surface tension of water-based inks to allow adhesion onto non-porous
19 substrates. The inclusion of surfactants in ink formulations however results in the
20 creation of a stable foam during mixing [Leach, 1988]. Water-based ink formulations
21 containing wax, surfactant, and defoamer additives were prepared and printed on
22 polyethylene film substrates. Standard test methods for adhesion, rub resistance, and
23 gloss were undertaken on the printed polyethylene films. Quantitative image analyses
24 were undertaken to quantify adhesion and rub resistance. Quantitative measurements
25 were also carried out to quantify gloss [Carlo, 2018]. Water-based ink [Wang, 2011;
26 Pang, 2014; Gong, 2014] mainly comprises the following components: starch, deionised
27 water, sodium hypochlorite, sodium hydroxide [Yang, 2014; Li, 2014; Chen, 2008],
28 polyvinyl alcohol, urea, nickel sulphate, colorant and auxiliary agents. Auxiliary agents
29 include defoamers, driers and surfactants. Water-based inks are considered
30 environmentally friendly within the packaging industry, and yet the water-based inks
31 currently used for printing still have notable disadvantages *i.e.* the complicated
32 preparation process and high cost, of a single use, water-based ink and pigment
33 [Recipe, 2015] and printing method. A recently patented water-based ink claimed to
34 have solved these problems. In view of these problems, recent patents have also
35 claimed to improve the adaptability of water-based inks [Zheng, 2016; Niu, 2016;
36 Zhonghao 2015], via the printing method and the type of pigment used. Water-based
37 inks have the advantage over solvent-based inks in that they are non-toxic, odourless
38 and non-polluting with respect to the environment. The inks are claimed to have good
39 bonding properties and their application is very broad *i.e.* they can be used for screen
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3 printing, flexography and gravure printing. Flexographic printing is widely used for
4 packaging applications and the properties required of the print to be fit for purpose,
5 with respect to the range of packaging applications and storage conditions can be
6 extremely diverse. The adhesion and flexibility of water-based inks to impervious
7 substrates is controlled by the selection of the polymer emulsion (Leach, 1993]. The
8 reason that latex is widely used as a pigment binder in paper coatings is that it
9 overcomes many dilemmas such as binder migration, print mottle, blister, water
10 retention and varied coating rheology (El-Sherbiny, 2014]. Semi-continuous emulsion
11 polymerisation, hetero-phase polymerisation with reactive surfactants, or by non-
12 seeded semi-batch emulsion polymerisation, were carried out for different applications
13 for this type of copolymer, such as binders for flexographic printing inks (El-Sherif,
14 2017]. Styrene-butyl acrylate copolymers were synthesised utilising different emulsifier
15 systems and modified with acrylamide (El-Sherif, 2018). The hydrophobic alkali-
16 soluble emulsion (HASE) was prepared using a semi-batch emulsion polymerisation
17 technique and evaluated as a binder for water based printing inks (Abd El-Wahab,
18 2019].

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27 The research reported in this manuscript focused on improving the mechanical
28 and physical properties of flexographic printing ink by using the prepared acrylic
29 copolymers emulsions, with different types of surfactant. A selection of the copolymers
30 were formulated with the presence of pigments, and other ingredients, to give water-
31 based flexographic inks, which were then characterised. The key aim of this work was
32 to prepare stable co-polymers based on acrylic monomers by an emulsion
33 polymerisation technique, to be used in the preparation of water-based flexographic
34 inks.

35 36 37 38 **Experimental**

39 40 **Materials**

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42 Styrene monomer (St), butyl acrylate (BuAc), acrylic acid (AA) and acrylamide (AAm),
43 supplied by Sigma-Aldrich Company, USA, were used as essential monomers.
44 Potassium persulphate (KPS), sodium acetate, nonylphenol ethoxylate (NP30) and
45 sodium lauryl sulphate (SLS) were supplied by Sigma-Aldrich Company, USA. Sodium
46 salt of fatty alcohol sulphate (supplied by Cognis, GmbH under the trade name
47 SULFOPON 12 G) was used as an anionic emulsifier. Anti-fungi (VINYZENE IT-4000
48 DIDP), dipropylene glycol methyl ether (Dowanol DPM) and anti-microbial (DOWICIL
49 QK-20), used in this research, were supplied by the DOW Chemical Company.
50 JONCRYL90 used was imported from BASF Chemical Company. The wetting agent
51 Surfynol 104 was imported from Muzing Company and the anti-foaming agent BYK 037

was provided by the BYK Company. Polyethylene wax used was produced by the Johnson Polymer Company and the Eljon Red 2RXs (yellow shade red pigment), the Barium Lithol Red (C.I. Pigment Red 49:1), and the DCC Pink Fb (blue shade red copper ferrocyanide pigment) were imported from the DCC Chemical Company.

Procedures

Pre-emulsion

Distilled water and emulsifiers St and BuAc were mixed in a flask equipped with a high speed homogeniser for 30 minutes. AA was then added to co-polymer during continuing homogenising.

Emulsion polymerisation of St/BuAc co-polymer (Snuparek, 2005)

The co-polymerisation of St and BuAc was carried out in a water bath, with the material being refluxed in a 1000ml three-necked flask equipped with a stirrer and having a gas inlet system. The oxygen was removed by purging nitrogen through the mixture for at least 30 minutes. Then, 80.5 ml of distilled water was introduced into a glass reactor with 2.2 g sodium lauryl sulphate (SLS) and SULFOPON 12 G as anionic surfactants. Then, 0.4 g of NP30 non-ionic surfactant was added and this was followed by 8 ml of distilled water and 0.1 g of potassium persulphate (KPS) as the initiator. The mixture was stirred under nitrogen atmosphere at 80°C. The SLS dissolved in distilled water (2.2 g in 59.6 g), was then mixed with different ratios of St and BAc according to polymer type, using vigorous stirring with the homogeniser at room temperature for 30 min. The 10% of obtained pre-emulsion was charged into the reactor for 30 min as a seeding stage, and then there was a 30 min wait before the 0.1 g KPS initiator, dissolved in 17 ml distilled water, was gradually added. The 2% AA and the 0.4 NP30 were mixed with the remaining 90% pre-emulsion, using the homogeniser, and this was charged into the reactor within a 3 hour period. The reaction was allowed to proceed under continuous stirring at 80 rpm and then the temperature was raised to 83 °C. At the end of the pre-emulsion addition, the temperature was raised from 83 to 85°C for 1 hour to complete the polymerisation reaction. The prepared emulsion was then cooled to 40°C and the pH adjusted to pH 8 using an aqueous solution of ammonia. Relevant details are represented in *Scheme 1 and Table I*.

Take in Table I

Take in Scheme 1

Latex formulation strategies

Using a different surfactant system

The latex systems containing monomer proportions of St/BuAc 25/25 weight ratio

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3 were set up using various proportions of emulsifier utilising SLS and NP30. The recipe
4 for the synthesis is given in *Table I*.
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6 **Preparation of water-based flexographic inks**

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8 Pigmented ink exists as a dispersion and the state of dispersion is dependent upon the
9 formulation and the process of manufacture. The normal method of measurement is
10 the grinding gauge consisting of a metal wedge graduated from 0 to 25 μm . Thus, the
11 ink is drawn down the wedge with a blade until pigment particles begin to form
12 scratches. This is then taken as the particle size (although more likely as an indication
13 of the size of pigment particle aggregates). Care should be taken that the test is
14 conducted prior to the addition of waxes etc. since these will interfere with the result.
15 Other indications of the dispersion state are gloss, colour development and
16 transparency and these are also monitored. Alterations in settings controlling the
17 conditions on milling equipment will give rise to variations in quality of dispersion and
18 careful monitoring will be needed to avoid batch variations. Chip-based inks are more
19 consistent from batch-to-batch than with other methods of high dispersion. Chipping
20 can, however, induce loss of flow due to the high dispersion state achieved. While
21 processing plays a major part in producing the dispersion, there is a considerable
22 influence and interdependence between the binder, pigment and solvent. All are
23 equally important to the final result, particularly with respect to the dispersion stability.
24 Finding the optimum conditions and formulation balance for maximising dispersion for
25 a particular product can be time consuming, and involve numerous experiments. There
26 is little doubt that bulk manufacture of single pigment bases, leading to fewer products
27 milled, is to be preferred since in this way only a few products have to be studied.
28 Stability of the dispersion is important, both with neat ink as supplied and diluted ink
29 as used on the press. The adhesion and flexibility of water-based inks to impervious
30 substrates is controlled by the selection of polymer emulsion. These are usually based
31 on acrylic resins and there are a wide range of types available. The softer, low- T_g (glass
32 transition temperature) polymers normally give the better adhesion and flexibility
33 properties when used on many film substrates, but these polymers have poor heat
34 resistance. Typical water-based flexographic ink formulations would contain the
35 following: 10 % of St/BuAc Co polymer, 18.4 % alkali soluble acrylic co-polymer, 51.94
36 % water, 0.36% antifoam, 0.1 % antibacterial, 2 % isopropyl alcohol, 3% polyethylene
37 wax emulsion, 0.2 % wetting agent and 14 % pigment. Recipes of flexographic ink
38 formulations containing the prepared nano-emulsion co-polymers are represented in
39 *Table II*. Following stirring to achieve a good dispersion, the pH value should be 8-9,
40 viscosity a 50-60 second measurement using Ford cup No. 4 at 25 °C, and grind should
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be 0-5 microns. The degree of dispersion was measured using a fineness of grind gauge. Also, water resistance and colour strength were measured.

Take in Table II.

Characterisation

a. Transmission electron microscopy (TEM)

The morphology of the copolymer particles was examined using transmission electron microscopy (TEM). The shape and particle size distribution of the nanoparticles were determined using a JEOL-JEM-1011 transmission electron microscope.

b. Gel permeation chromatography (GPC)

The weight-average (M_w) and number-average (M_n) molecular weights were determined using an Agilent 1100 series gel permeation chromatography (GPC) system, equipped with a G1362A refractive index detector with 100-104-105 Å^o ultra styragel columns. Polystyrene was used as a standard. THF was used as an eluent with a flow rate of 1 ml/min.

c. Coagulum

Coagulum of the polymer is generally done on a 100 g lot, and are a simple gravimetric determination of that percent retained on a 100 mesh. And the coagulum collected were determined according to ASTM D4828.

d. Density

A pycnometer, having a volume of 50 cm³ is utilised to determine the density of the emulsions, at 25 °C. Thickness of polymer is more noteworthy than 1 g/cm like all the water-based latex. Exceptional hydrometers are likewise utilised for fast assurance of latex thickness. Thickness esteem is essential for the vehicle and shipment of the fabricated latex, it has less logical disadvantages (El-Sherif .H. M, 2018) .

e. Thermo gravimetric analysis (TGA)

TGA analysis was performed using a Shimadzu TGA-50 thermo gravimetric analyser (Columbia, EUA) using a nitrogen atmosphere and a heating rate of 10 °C/min over the temperature range between room temperature and 600 °C.

f. Fourier transform infrared (FT-IR)

The FT-IR analysis of the dried polymer samples was carried out using a JASCO FTIR 6100 in the range of 4000–400 cm⁻¹.

g. Viscosity

The consistency of the flexographic inks was assessed by utilising a Ford viscosity cup

number 4 at 25 °C by filling the cup with the fluid under test, and recording the time taken for the material to run through the standard openings (ASTM D 1200).

h. Degree of dispersion

This test method describes the procedure for determining the fineness of grind of the printing inks, using a NPIRI grindometer. It evaluates the size of the largest particles in finished dispersion but not the average particle size, or the concentration of size. This test method is applicable to any dispersion that is fine enough to fall within the 0–25 µm range of the specified grind gauge. With a minor variation in procedure, it is applicable to both paste (non-volatile) and liquid (volatile) inks (ASTM D 1210).

i. Colour density

Inks formulated from the prepared copolymers were applied onto white paper using an anilox applicator. The colour density of the printed sample was then measured using an ultra-scan pro spectrophotometer.

Results and discussion

This work was expected to provide an ideal 50% emulsion for modern day application in water-based flexographic inks. Thus, a significant number of trials were undertaken in an attempt to create an ideal performance product from (St/BuAc) emulsion co-polymer, having stable properties for a time-frame of 6 months. Also, with an aim to achieve a low coagulum of (St/BuAc) emulsion co-polymer, the prepared polymers were investigated by testing for: solid content %, wet coagulum 100 mesh (g/l), particle size (nm), viscosity (cP), molecular weight, hardness, adhesion and pH. Characterisation of these systems was undertaken using TEM, TGA and FT-IR spectroscopy.

Characteristics of the prepared emulsion copolymers of St-co-BuAc

The physical, chemical and mechanical properties of the prepared (St/BuAc) utilizing SULFOPON 12 G , SLS group (I), group (II) and group (III) were inspected using global standards and the outcomes are outlined in Table 4. In addition, the pencil hardness test, the bowing trial of the film, and the attachment to various substrates, were analysed and recorded in *Table III*.

Take in Table III.

Effect of SLS, SULFOPON 12G and NP30 surfactant system on solid content of Group I, Group II and Group III (Hu, 2011; Hou-Hsein, 2007; Chorng-Shyan, 1984; Samira 2012; Abd El-Wahab, 2018)

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3 The solid content of the prepared copolymers was determined gravimetrically. It was
4 found that solid content depends mainly on the polymerisation rate and reaction
5 conditions and less so on monomer composition. *Figure 1*, shows the relationship
6 between changing the surfactant (SLS, SULFOPON 12G) and the solid content of the
7 prepared co-polymer. It is clear from the data that group I had the maximum solid
8 content, while group III had the minimum solid content. Also, we can conclude that as
9 the hydrophobic monomer increased, the solid content decreased for all the groups. In
10 addition, the values of the solid content slightly increased by altering the type of anionic
11 surfactant SULFOPON 12 G, sodium lauryl sulphate (SLS) (A > B), (C>D) and (E>F).
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16 **Take in Figure 1**

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18 *Effect of SLS, SULFOPON 12G and NP30 surfactant system on the viscosity of Group*
19 *I, Group II and Group III*
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21 The viscosity of a latex at any given total solids content decreases with increasing
22 particle size and with increased broadening of the particle-size distribution. *Figure 2*
23 shows the relationship between changing the surfactant (SLS, SULFOPON 12G) and the
24 viscosity of the prepared co-polymer. It is clear from the data that Group III has a
25 higher viscosity than Group I and Group II. Also, we can conclude that as the
26 hydrophobic monomer increases, the viscosity also increases. The viscosity value also
27 increases slightly by changing the type of anionic surfactant SULFOPON 12 G, SLS. We
28 can conclude that Group III has a better emulsification system compared to the
29 emulsification system of Group II and Group III. Low viscosity may reduce the film
30 thickness of the flexographic inks and may decrease the spreading of the ink on the
31 surface.
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37 **Take in Figure 2.**

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39 *Effect of SLS, SULFOPON 12G and NP30 surfactant system on the particle size of*
40 *Group I, Group II and Group III.*
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42 Particles in an ink must be small to allow a film to develop around the beads in the
43 scattering stage. Diminished molecule measure allows more particles to be surrounded
44 at the limit, yielding a more stable emulsion. The size of emulsion particles is affected
45 directly by the emulsification system, conditions of polymerisation, and the relationship
46 between the co-monomers. As illustrated in *Figure 3*, it is clear that Group I has the
47 largest size of emulsion particles, while Group II and Group III has emulsion particles
48 of a smaller size. Also, we can conclude from Figure 3 that as the hydrophobic
49 monomer increases, the size of the particles decreases. Thus, the large particles allow
50 for film development in a flexographic ink formulation.
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56 **Take in Figure 3.**

Effect of SLS, SULFOPON 12G and NP30 surfactant system on the molecular weight and molecular weight distribution of Group I, Group II and Group III (Shi, 2013)

Molecular weight is an important factor that affects many polymer characteristics. The Mwt of the prepared polymers has a direct relationship with particle size. The range of molecular weight average Mwt is between 2.2 and 2.9×10^5 . The range of molecular weight distribution Mn is between Mn 6 and 7.05×10^4 . Obviously, the co-polymer A (Mwt = 2.9526×10^5 , Mn = 6.0289×10^4) has a higher molecular weight and narrower molecular weight distribution than co-polymer B (Mwt = 2.2435×10^5 , Mn = 7.0507×10^4). This implies that the emulsification arrangement of Group I, which contains SLS, gives good emulsion properties.

FT-IR spectroscopy of the prepared copolymer Group I, Group II and Group III

In infrared spectroscopy, the resulting spectrum represents the wavenumber and transmission, creating a molecular fingerprint of the sample. The FT-IR spectrum shown in *Figure 4, of Sample A, Group I*, illustrates the following functional groups: the carbonyl group C=O double bonds appear in the region of 1727.7 cm^{-1} , the band at 2958 cm^{-1} is specific for the aliphatic hydrocarbon of butyl acrylate and vinyl acetate, whereas the band at 3340 cm^{-1} is due to a styrene ring. The FT-IR spectrum *Figure 4 of Sample F, Group II*, illustrates the following groups: the carbonyl group C=O double bonds appear in the region of 1727.6 cm^{-1} , the band at 2957 cm^{-1} is specific for the aliphatic hydrocarbon of butyl acrylate and vinyl acetate, whereas the band at 3343 cm^{-1} is due to a styrene ring.

Take in Figure 4.

The FT-IR range of the latex demonstrates that styrene and butyl acrylate participated in the copolymerisation response and that styrene acrylate copolymerisation latex was prepared.

Thermo gravimetric analysis (TGA) of the prepared copolymer Group I, Group II and Group III

The TGA thermograms of the polymer samples, together with the derivative thermograms (dW/dT drawn with respect to temperature) are shown in *Figures 5a, 5c and 5e*. The curves show that the degradation is completed in a single step. Comparing the TGA thermograms of the co-polymer latexes films, slight differences were noticed. They all exhibited the same final decomposition temperature ($500 \text{ }^\circ\text{C}$) but had different weight loss%. The initial decomposition temperature was observed at 385, 375 and $370 \text{ }^\circ\text{C}$, respectively. The final decomposition temperature was at 408, 400 and $404 \text{ }^\circ\text{C}$, respectively. Weight losses were 74.5%, 73.3% and 78.50%, respectively. The peak temperature of the main degradation step for Sample A has shifted to a higher

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3 temperature compared to Samples C and E. This is an indication that the prepared
4 Polymer A is more stable than Polymers C and E. This indicates that the chemical
5 structure plays an important role in the thermal decomposition (Pashaei, 2010).
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8 **Take in Figures 5a, 5c and 5e.**

9 *Transmission electron microscopy (TEM) of the prepared polymers*

10 It has been reported that the hydrophobic/hydrophilic character of the monomer(s)
11 used in emulsion polymerisation has a decisive influence on particle morphology, with
12 the chemical structure of the emulsifier also being a key factor. For example, planar
13 polymeric microstructures can be prepared by emulsion polymerisation using
14 polymerisable branched emulsifier [Chi-Jung, 2006]. According to some authors,
15 macro molecules with an increase in hydrophilicity facilitate carboxyl ionisation,
16 resulting in higher particle swelling (increased viscosity), and particles with a high
17 content of AA being completely solubilised (Lovell, 1997; Zhao, 2004]. The morphology
18 of the particles obtained during the different experiments is illustrated by the electron
19 micrographs shown in **Figure 6**. These micrographs show the effect of the
20 emulsification system and the relationship between styrene and butyl acrylate on the
21 particle size and shape of the prepared emulsion lattices. By changing the emulsifier
22 system, from SULFOPON 12G and NP30 to SLS and NP30, the viscosity of the medium
23 increases relatively and it becomes difficult for the two polymer phases to rearrange
24 themselves to a thermodynamically stable morphology.
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33 **Take in Figures 6.**

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35 The TEM electron micrographs demonstrate that the width of most particles
36 arranged by emulsification framework for Group I and Group II are between 120 and
37 150 nm, as indicated in *Figure 6*, and have a limited dispersion. The measurement of
38 most particles arranged by emulsification framework for Group III were between 90
39 and 110 nm, as indicated in *Figure 7* and have wider dispersion. All particles consist of
40 a St/BuAc core and an acrylic acid shell. It is obvious from the micrographs that the
41 particles are spherical in shape and have no distortion.
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45 **Take in Figures 7.**

46 *Storage stability test*

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48 As a multifunctional additive, hydrophobic alkali swellable emulsion (HASE) makes it
49 possible to reduce costs and still obtain performance equal to or better than that of
50 similar ink formulations containing thickener. In order to check the long-term stability
51 of the ink and its dependence on nano-emulsion binder, it is recommended to store the
52 ink at elevated temperatures (50-60 °C) for two weeks and to check viscosity and colour
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3 strength afterwards. Insufficient storage stability might lead to flocculation. This is the
4 general term for weakly interaction between associative thickener and binder because
5 the main ingredient for anti-settling in ink formula is acrylic associative thickener as
6 shown in Table III and the latex has large particle size. Settlement leads to loss of gloss,
7 loss of colour strength and settling tendency.
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10 **Preparation and evaluation of water-based flexographic ink formulations based on** 11 **the prepared St/BuAc emulsion co-polymer**

12 *Preparation of water-based flexographic inks (Abd El-Wahab, 2019)*

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14 The adhesion and flexibility of water-based inks to impervious substrates is controlled
15 by the selection of the polymer emulsion. These emulsions are usually based on acrylic
16 resins and there are a wide range of types available. The softer, low- T_g (glass transition
17 temperature) polymers normally give the best properties for adhesion and flexibility
18 when used on many film substrates, although these polymers have poor heat resistance.
19 Typical water-based flexographic ink formulations contain the following components:
20 10% of St/BuAc co polymer, 18.4% Alkali soluble acrylic co-polymer, 51.94% water, 0.36
21 antifoam, 0.1% antibacterial, 2% isopropyl alcohol, 3% polyethylene wax emulsion, 0.2%
22 wetting agent and 14% pigment. After stirring until a good dispersion was obtained,
23 the pH value was 8 - 9, viscosity was 50-60 seconds following measuring with a Ford
24 cup No. 4 at 25 °C, and grinding was 0-5 microns. The degree of dispersion was
25 measured using a fineness of grind gauge. In addition, water resistance and colour
26 strength were also measured.
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35 **Viscosity, water resistance and Colour density of the prepared flexographic ink based** 36 **on the nano-copolymers as a binder**

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38 Samples of the prepared nano-copolymers were incorporated into emulsion water-
39 based ink formulations as a binder and evaluated. The samples were compared with a
40 commercial trade mark reference co-polymer (Joncryl). The results obtained for water
41 resistance, Colour density and viscosity, based on the nano-copolymers as a binder,
42 showed improved values when compared with the commercial reference sample
43 (Joncryl). The data are shown in Table IV. Flexographic water-based inks are used for
44 printing on paper, carvings and duplexes and are used in the manufacture of cans and
45 carton boxes. Also, there are thermoplastic inks used for printing on plastics and
46 textiles. Water-resistance in the printed product was assessed by using St/BuAc
47 emulsion co-polymer. The co-polymer emulsions were incorporated into a standard
48 formulation for water-based flexographic inks and compared with a co-polymer
49 emulsion used in industry as previously illustrated in *Table III*. The prepared water-
50 based flexographic inks were tested for viscosity, pH, grinding and water resistance, as
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3 shown in *Table IV*. Table IV summarises the results obtained for the water-based
4 flexographic inks using Group I, Group II, Group III and Joncryl 90 in standard ink
5 formulations.
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8 Take in Table IV.
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10 *Viscosity*

11 Based on the results tabulated in Table VI, it is clear that the viscosity of the prepared
12 polymers in Group I and Group II have slightly lower viscosity values when compared
13 to the trademark co-polymer (Joncryl). This lower viscosity may reduce the film
14 thickness of the flexographic inks and may also decrease the ink spread on the surface.
15 This could reduce resistance to staining and make cleaning easier. It is clear from the
16 data that Group III has a higher viscosity than Groups I and II. Also, we can conclude
17 that as the hydrophobic monomer increases the viscosity increases .
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22 *Water resistance*

23 The addition and the type of copolymer balance the water-resistance in the printed
24 product. After an adequate amount of flexographic ink has been applied on paper by a
25 hand roller, the paper is immersed into water at 25 °C and left for 30 seconds.
26 Observation is then made to see whether the ink put down lifts from the paper or not.
27 As shown in *Figure 8*, three printed samples using different formulations of nano-
28 copolymer (St-BuAc), A, B and C representing Sample A Group I, Sample C Group II
29 and Sample E Group III were immersed in distilled water at 25 °C for 30 seconds. The
30 nano Co-polymer (St/BuAc) can sharply enhance the water resistance of the printed
31 paper due to its strong hydrophobicity. Thus, as the hydrophobic monomer increases
32 in the copolymer, the water resistance also increases as well. Available literature
33 confirms that the hard monomer St/BuAc is used for its hardness and water resistance
34 properties (Zeqin 2013).
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41 **Take in Figures 8.**
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45 *Colour density*

46 The Colour density of a flexographic print will be determined by the ink film thickness
47 applied to the substrate and the type and concentration of colorant used in the
48 formulation. The wet ink film thickness will, in practice, vary over a wide range of
49 approximately 2-15 µm, dependent upon press, metering, substrate and, not the least,
50 the working practice of the printer. On average, the film thickness is lower than that
51 achieved with gravure printing and a stronger ink is required to obtain satisfactory
52 coverage. In general, the values of colour strength are slightly increased by changing
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3 the type of anionic surfactant SULFOPON 12 G, SLS. Also, as the hydrophobic
4 monomer increases, so does the colour density. The superior three samples are Samples
5 A, C and E. The colour measurements were determined using a Hunter Lab Ultra Scan
6 Spectrophotometer. The L axis runs from top to bottom. The maximum for L is 100,
7 which would be a perfect reflecting diffuser. The minimum for L would be zero, which
8 would be black. The (a) and (b) axes have no specific numerical limits. Positive (a) is
9 red, negative (a) is green, positive (b) is yellow and negative (b) is blue. By changing the
10 type of anionic surfactant SULFOPON 12 G, SLS, the colour strength increases slightly,
11 as does the density (shown in *Table IV*).

12
13 Print quality is expressed by image sharpness, colour, tone, gloss, print density,
14 legibility and the uniformity of these properties (Hussain, 2010]. The specular gloss
15 relies on the surface structure and porosity of the substrate, while the print density is
16 evidence of a thicker ink layer. This is a consequence of the wider coating network and
17 the stronger bonding between the base paper and the ink (Gigac, 2011].

18
19 With respect to gloss and density, the size and shape of the pigment particles
20 have a real impact on both the coating and the printing processes. The gloss of a coated
21 sheet is reliant on the coating covering the fibres and on the roughness of that coating.
22 The latter has a unified two prime component that produces the surface topography
23 and consequently its gloss. Firstly, the pigment particles contribute to the small-scale
24 micro-roughness due to their size and shape, and the mode by which they pack
25 together on the surface of the paper. Secondly, the base paper fibres just beneath the
26 coating, impact the surface roughness, particularly at low coat weights since a low
27 amount of coating does not entirely match fibre size and shape.

28
29 Regarding the print quality of coating formula utilising Group I based on **(St- co-
30 BuAc)** nano-emulsion binder, it was observed that there was an average gloss reading
31 of 42-53 for the printing process. Print gloss and print density were directly correlated.
32 An explanation could be that low print gloss is evidence of a rough print surface, which
33 indicates that when print density is low, poor quality image and colour reproduction is
34 obtained (Sonmez, 2011]. Also, based on the tabulated results, the type of surfactant
35 can play an important role on the final properties. It can be observed that changing the
36 type of anionic surfactant SULFOPON 12 G, SLS, leads to a slight increase in colour
37 strength, together with density. So as we mentioned above all the mechanical and
38 physical characteristics obtained of water base flexo inks using group (I), group (II)
39 and group (III) based on **(St- co-BuAc)** copolymer are tabulated in *table IV*.

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Conclusions

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3 It is clear that the Group I have the maximum solid content, while Group III have the
4 minimum solid content. It can conclude that as the hydrophobic monomer increased
5 the viscosity was increased. The solid contents of the prepared (St- co-BuAc) were
6 determined gravimetrically. The viscosity value also increased slightly by changing in
7 the type of anionic surfactant SULFOPON 12 G, SLS. It is clear from the data that
8 Group III had greater viscosity than Group I and Group II. It can be concluded that
9 Group III have a better emulsification system compared to the emulsification systems of
10 Group II and Group III. The change in the surfactant (SLS, SULFOPON 12G) type lead
11 to change in the viscosity of the prepared co-polymer, as well as, the values of the solid
12 content slightly increased by altering the type of anionic surfactant SULFOPON 12 G,
13 SLS (A > B), (C>D) and (E>F). It was found that the solid content mainly depends on
14 the polymerisation reaction conditions. Clearly, the co-polymer A has a higher
15 molecular weight and a narrower molecular weight distribution than co-polymer B.
16 The viscosity of a latex at any given total solids content decreases with increasing
17 particle size and with increased broadening of the particle-size distribution. It was
18 observed that the average gloss 42-53 for the printing process while its print density
19 was of 2.04 -2.37. Print gloss and print density were directly correlated. Particle size
20 specifically affects the physical solidness of emulsions.
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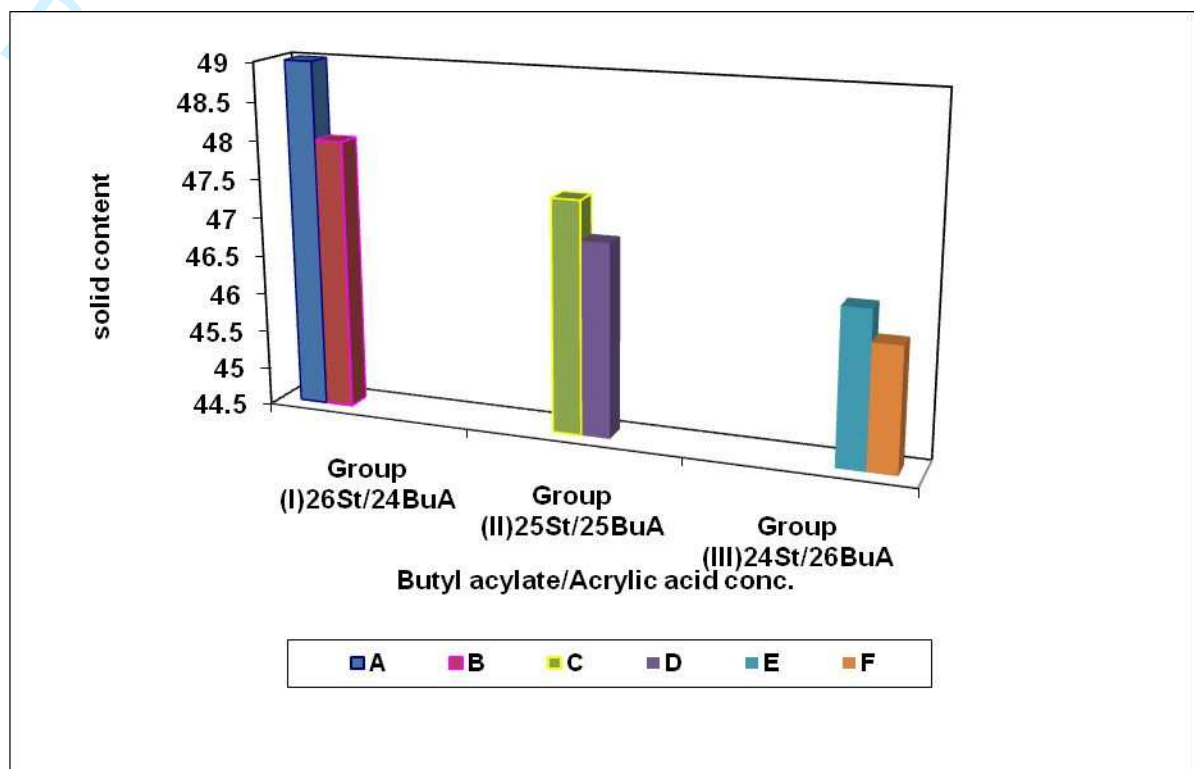


Fig. (1): Effect of changing surfactant systems and butyl acrylate/acrylic acid on solid content

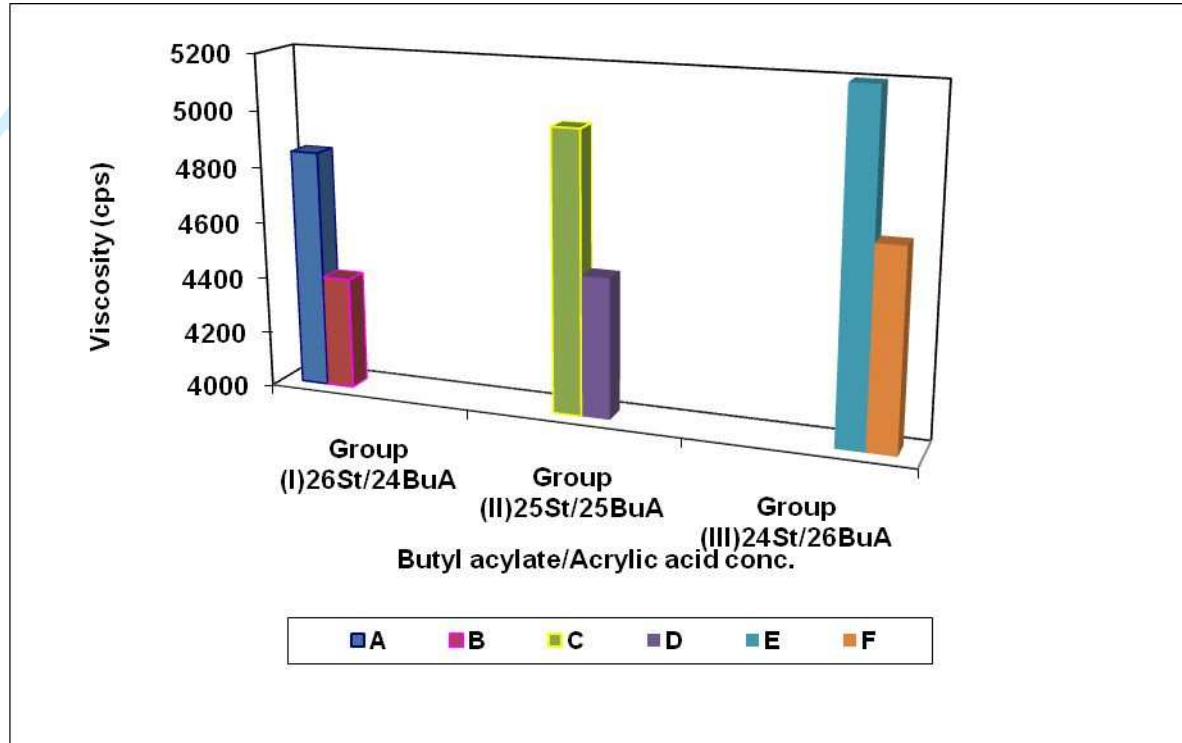


Fig. (2): Effect of changing surfactant systems and butyl acrylate/acrylic acid on viscosity

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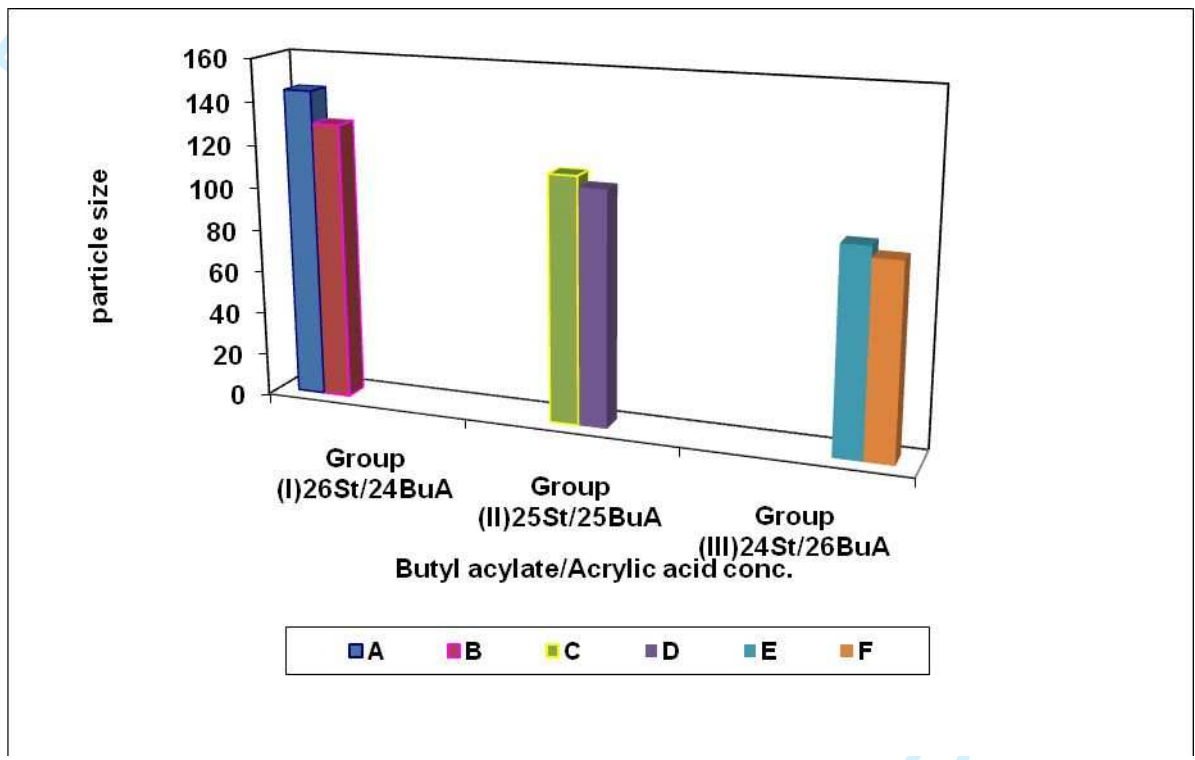
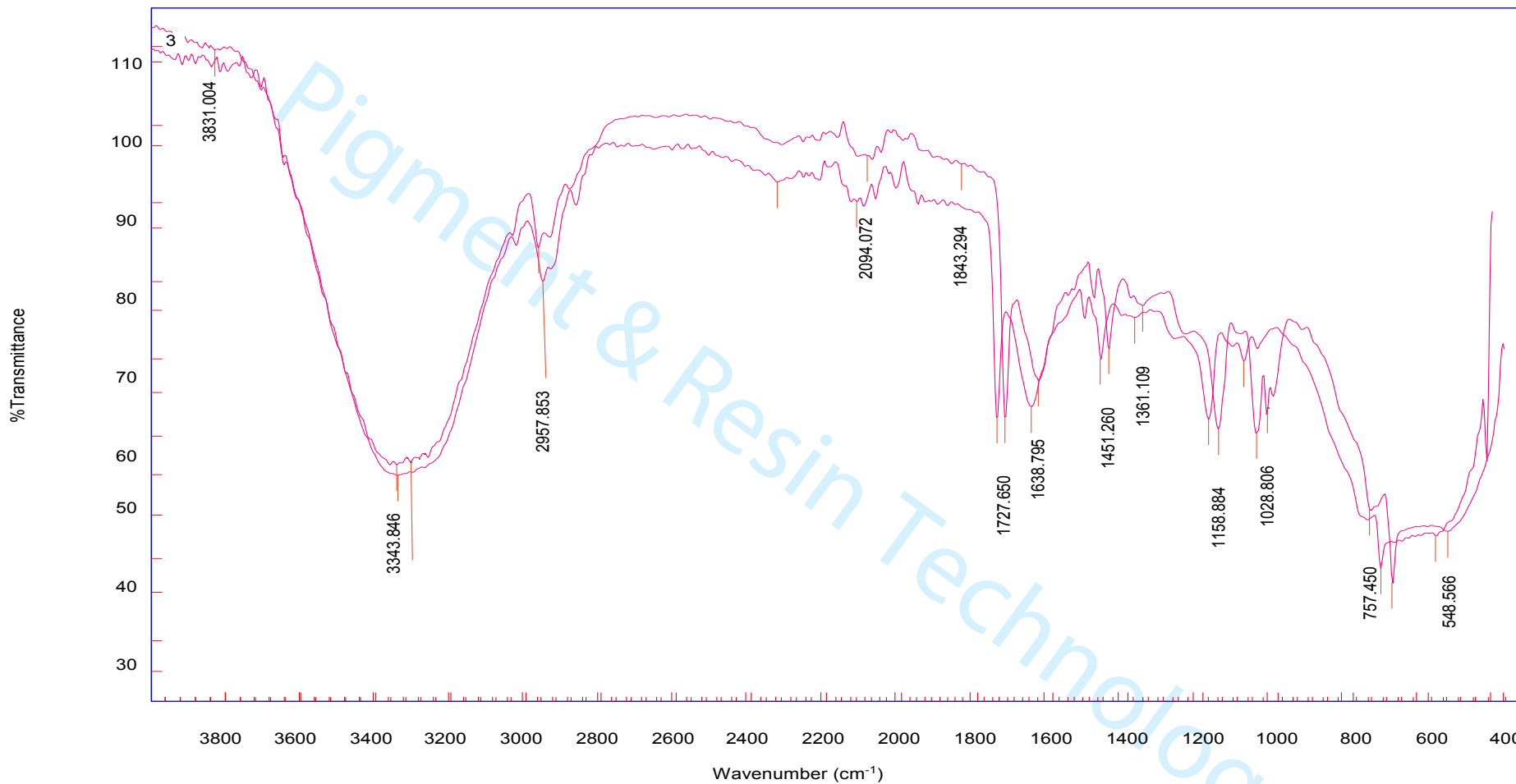


Fig. (3): Effect of changing surfactant systems and butyl acrylate/acrylic acid on particle size

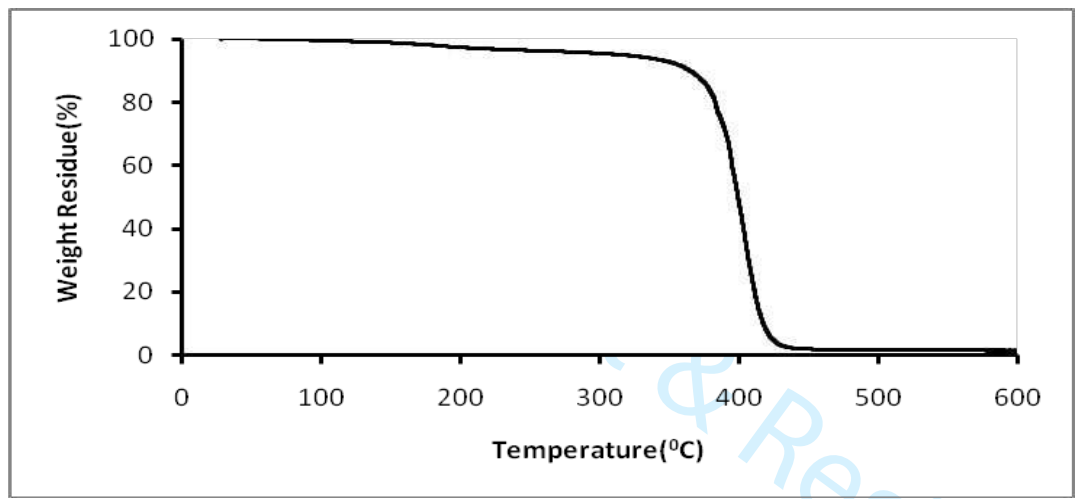
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2 **Samples (A&F)**
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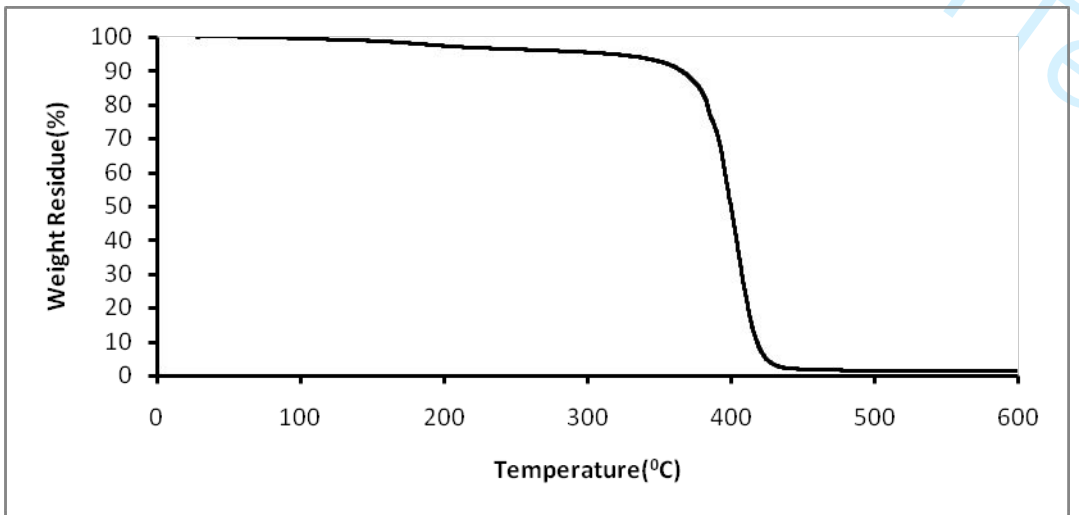
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37 **Figure (4): FT-IR spectra of styrene – butyl acrylate copolymer Group I sample A and Group III sample F.**
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Sample A



Sample C



Sample E

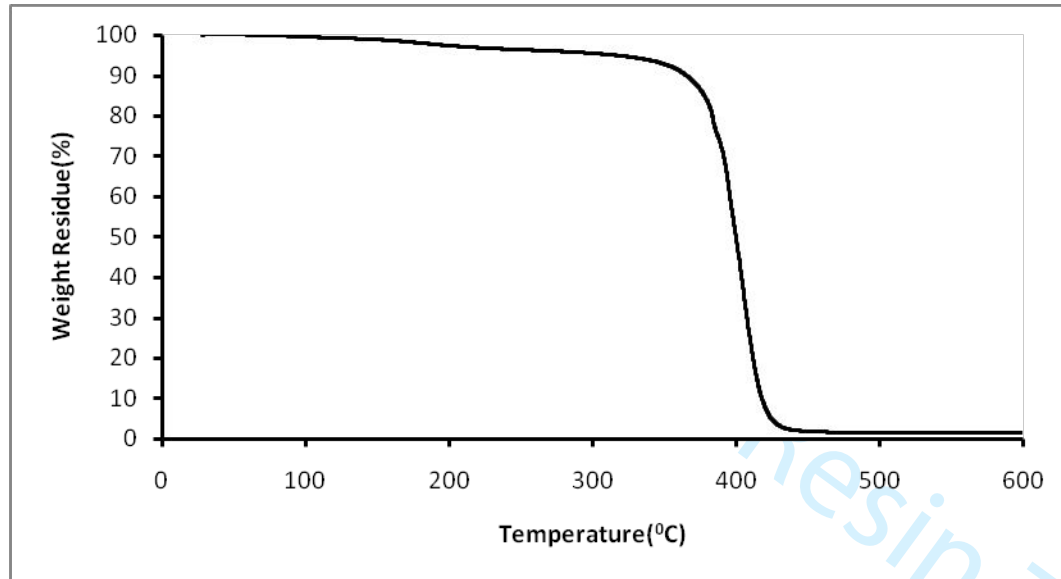


Figure (5): TGA curve for copolymers A,C and E /mass loss versus temperature

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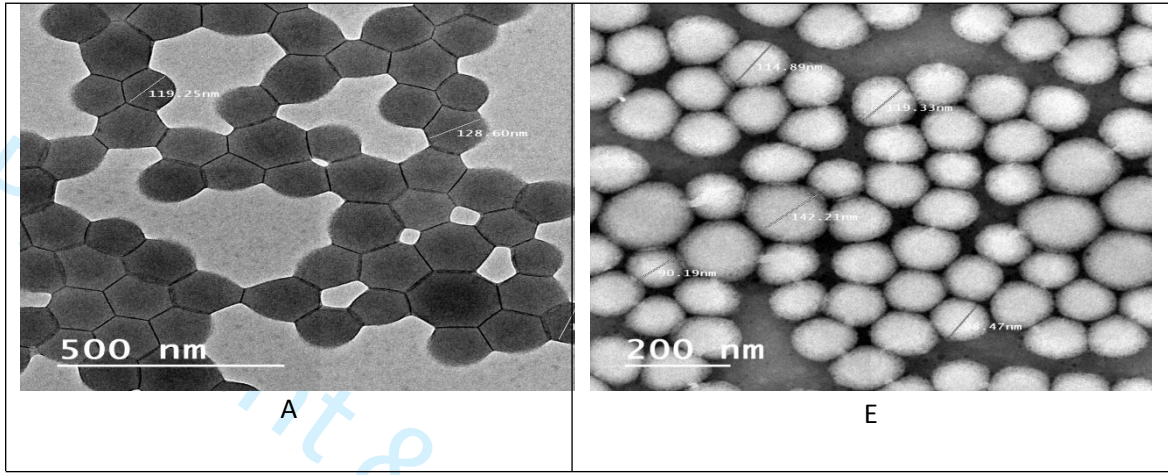


Figure (6): Transmission electron micrographs of the prepared emulsion copolymer sample A Group I (avg.120nm) and sample E Group III (avg.130nm).

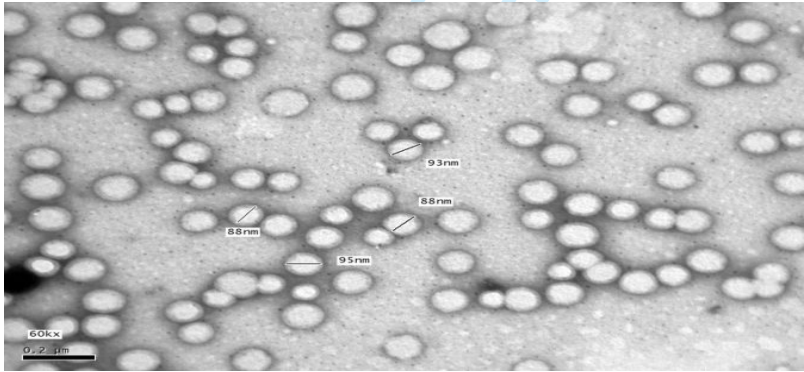
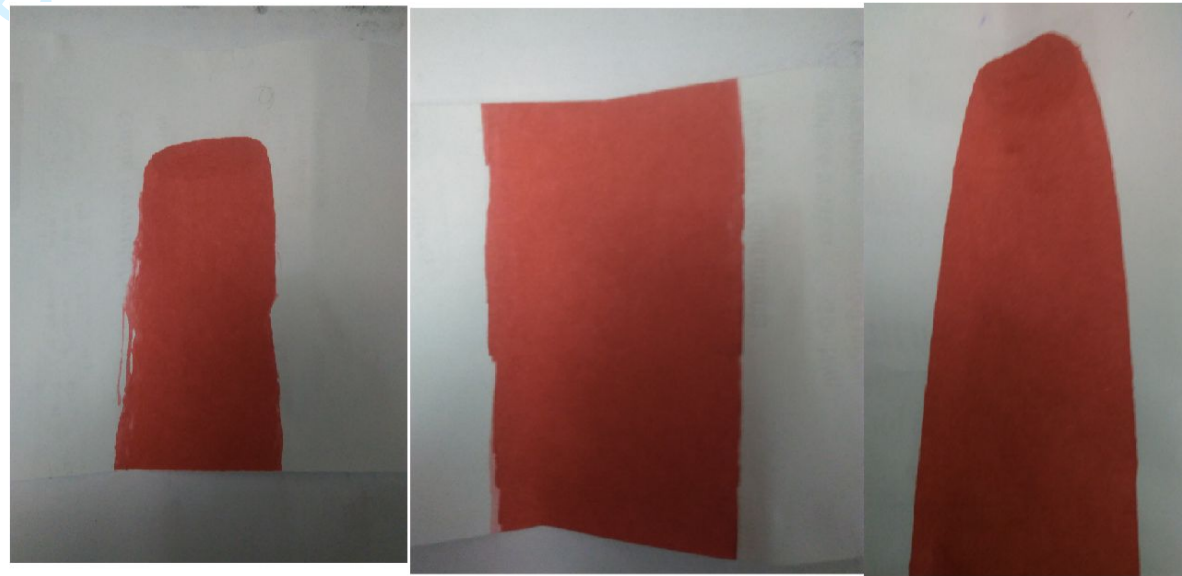


Figure (7): Transmission electron micrograph of the prepared emulsion copolymer sample B Group II (avg.90nm) .



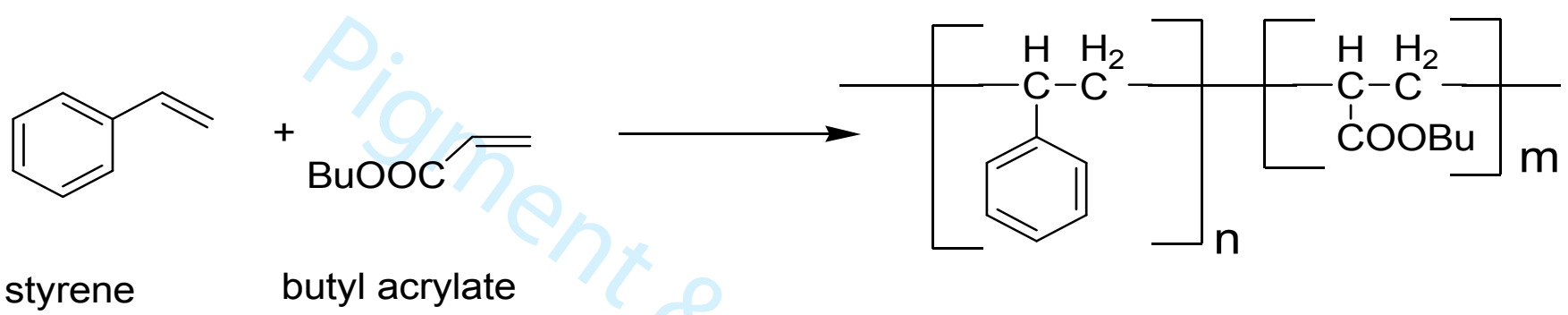
Sample (A)

Sample (C)

Sample (E)

Figure 8: Water resistance photo-macrograph of water-based flexographic ink samples using sample A group I , sample C group II and sample E group III

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Scheme (1): Chemical structure of the prepared styrene butyl acrylate (St/BuAc) nanoemulsion copolymer

Table I: Recipe for different ratios of St/ BuAc emulsion co-polymer containing of SLS, SULFOPON 12 G as anionic surfactant, NP30 as non ionic surfactant:

Component*	Group (I)		Group (II)		Group (III)	
	A	B	C	D	E	F
Styrene	26	26	25	25	24	24
Butyl acrylate	24	24	25	25	26	26
Sodium lauryl sulphate (SLS)	2.2	0	2.2	0	2.2	0
SULFOPON 12 G	0	2.2	0	2.2	0	2.2
NP30	0.4	0.4	0.4	0.4	0.4	0.4
Dest.H ₂ O	48	48	48	48	48	48

*All ingredients weighted in grams.

The above recipe contains pot.persulphate as free radical initiator (0.2 g), acrylamide monomer (0.1) and Acrylic Acid (2 g). Temp 80°C, pH 8.5 .

Table II: Recipes of Flexographic Ink formulations containing the prepared nano-emulsion co and terpolymers:

Ingredients*	Group (I)		Group (II)		Group (III)	
	A	B	C	D	E	F
St/ BuAc CO-polymer	10	10	10	10	10	10
Pigment red 49:1	4.8	4.8	4.8	4.8	4.8	4.8
Pigment red 81	0.2	0.2	0.2	0.2	0.2	0.2
Pigment red 2	9	9	9	9	9	9
Alkali soluble acrylic co-polymer	18.4	18.4	18.4	18.4	18.4	18.4
Antifoam	0.36	0.36	0.36	0.36	0.36	0.36
Antibacteria	0.1	0.1	0.1	0.1	0.1	0.1
Wetting agent	0.2	0.2	0.2	0.2	0.2	0.2
PE wax emulsion	3	3	3	3	3	3
Isopropyl alcohol	2	2	2	2	2	2
Water	51.94	51.94	51.94	51.94	51.94	51.94

*All ingredients weighted in grams.

Table III: Characteristics of the prepared of (St- co-BuAc) nanoemulsion copolymer.

Properties	Standard	Group (I)		Group (II)		Group (III)	
		A	B	C	D	E	F
pH	ASTM	7.5	7.5	7.4	7.5	8	7.8
Solid Content (%)	D2369	49	48	47.5	47	46.5	46.1
Particle size (nm)	TEM	145	130	115	110	95	90
Brookfield Viscosity RVT #50 rpm, (cps)	D 2196	4850	4400	5000	4500	5200	4700
Molecular weight	Mn*10 ⁴ (g/mole)	7.05	6.62	6.45	6.3	6.2	6
	Mw*10 ⁵ (g/mole)	2.9	2.7	2.6	2.5	2.4	2.2

Table IV : Mechanical and physical characteristics obtained of water base flexo inks using group (I), group (II) and group (III) based on (St- co-BuAc) copolymer.

	standard formulation by (JONCRYL 90)	Group (I)		Group (II)		Group (III)	
		A	B	C	D	E	F
Viscosity (Sec) Ford. Cup No.4 at 25°C	60	53	55	54	56	60	60
PH	9	9	9	9	9	9	9
Water Resistance	Excellent	Bad	Bad	Good	Good	Excellent	Excellent
Grinding	5	5	5	5	5	5	5
Gloss	42	42	42	46	48	50	53
Color Strength (density)	2	2.2	2.04	2.32	2.1	2.37	2.19