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Polymeric materials as pour point depressant for Egyptian waxy crude oil

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ABSTRACT

The relation between the temperature of waxy crude oils pipelining and pour point is a well-established phenomenon. Different techniques have been applied to reduce the problems due to crystallization of saturated hydrocarbons during production and transportation of waxy crude oils. Use of pour point depressant (PPD) is preferred economically and effect cost saving. In this work, Poly-methyl acrylic acid hydrazide, modified N-Salicylidene polymer and tin chloride complex was synthesis and characterized by SEM, FTIR, BET and TGA. PPD was evaluated as flow improver in different types of Egyptian waxy crude oils, Alamin (light crude oil) and Qarun (heavy crude oil).The results showed good performance in improving the pour point temperature depending on the composition and the location of waxy crude oil. The optimum amount of PPD for three types of depressants is 500 ppm for both types of Egyptian waxy crude oils.

Keywords: Pour point depressant, flow improver, waxy crude oil.

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INTRODUCTION

Crude oils consist of linear and branch wax containing a large amount of wax and high viscosities. It often requires specific conditions pressure (i.e. yield stress) to initiate the flow for pipelining. Wax deposition is a serious problem in crude oil production and processing. Waxy crude oils contain significant amount of paraffin waxes and asphaltenes and these components are the main problems in reservoir, production tubing and transportation through pipe (Mahto and Kumar, 2013). Resins are not considered as a deposit on their own, but it deposit with asphaltenes together (Sharma et al., 2012; Venkatesan et al., 2005). Crude oils will stop flowing in a pipeline and following a sudden shutdown of pumping operation, and increase pressure drop across the pipe if the ambient temperature is below its pour point (Hemant and Dinakar, 2006). Several treatments are available to solve this problem like mechanical solutions play an important role such as passing pigs removal, steam tracing and thermal insulation of pipelines can be used (Galloway, 1992; Sarmento et al., 2014). In addition internal coating of pipes with antistick material, application to modify the crystals and the use of chemical additives, such as organic inhibitors and pour point depressants (PPD), which drops the deposition of waxes and the pour point of the crude oil as well (Lucas et al., 2015; Bucaram, 1967). Pretreatment with flow improver (FI) additives is a solution for transportation of waxy crudes in pipelines. Flow improver additives, also known as pour point depressants (PPD), can reduce the growth of wax crystals and form smaller crystals of higher volume to surface ratio. Due to this change in crystalline shape, the ability of the wax crystals to grow and interlock is reduced. The combination of these effects lowers the pour point, viscosity, and yield stress considerably, making transportation much easier (Holder and Winkler, 1965a; Holder and Winkler, 1965b). In nature, flow improvers are very selective. The affectivity of additives varied according to the type of crude (Sifferman, 1979).

Some depressants reduction of viscosity and strength do not effect on pour point and besides, the rheological parameters such as viscosity, pour point, and yield stress of the treated crude may regain the original gel structure of the untreated crude, in case of a sudden shut down for a period. However, when an exact fit can be developed, polymeric additives can be remarkably effective in depressing the pour point of waxy crude (Irani and Zajac, 1982). So, we can say that wax content is responsible for flow problems: the crude which has a high quantity of wax contains higher the wax contain, the greater are the flow problems. However, there is no relation between pour point and wax content. Other constituents in the crude oil (i.e., asphaltene, resin, lighter distillates, etc.) also effects on flow behavior. For example, crude oils with high asphaltene contents are also highly viscous (Wardhaugh and Boger, 1987). Moreover, asphaltenes the main factor of waxes formation. They focus on the surface of the wax crystal. In the present work, synthesis of three new pour point depressant to play an important role to drop down pour point for two Egyptian crude oils were selected on the basis of wax and asphaltene contents to minimize wax deposition due to temperature reduction during crude transportation.

EXPERIMENTAL

Materials

Methyl acrylate monomer (Merck, assay ≥ 99.5%), Potassium bromate, Sodium hydrogen sulfite, Di-methyl sulfoxide (Merck, assay 99%), Hydrazine hydrates (ALPHA CHEM, 99%), methanol (SOMATCO, assay ≥ 99.7%), Salsicylaldehyde (Merck, assay 98%), dimethyl tin dichloride (assay 97%), chloroform all chemicals used were analytical grade. Two different types of Egyptian waxy crude oils were used for evaluating the performance of synthesis polymer additive.

Physical characteristics of crude oils

Two paraffinic crude oils are studied in this work, that is, Alamin and Qarun crude oils. Table 1 represents physical characterizations of these crudes.

Table 1. Physical properties of Egyptian waxy crude oils.

Properties	Alamin crude oil	Qarun crude oil	ASTM methods
API	39.6	37.6	ASTM D287
Specific gravity, 60/60°F	0.825	0.8361	ASTM D-1298
Pour point, °C	6	27	ASTM D-97
Wax content, wt%	3.2	5.6	ASTM D5442
Asphaltene content, wt %	0.61	1.3	ASTM D6560
Water, vol %	0.01	0.01	ASTM D4006

METHODS

Synthesis of poly methyl acrylate (based polymer)

Polymerization of methyl acrylate monomer is carried out using potassium bromate, sodium hydrogen sulfite as initiators. These contents are mixed together in flask till particles starts to grow and quickly turn to milky appearance. Then, the milky emulsion is added to sodium chloride to coagulate the polymer then washed by water to remove unreacted monomer the removed by tweezer.

Synthesis of poly-methyl acrylic acid hydrazide (polymerization reaction)

Polymerization of methyl acrylate monomer is carried out using Potassium bromate, Sodium hydrogen sulfite to obtain poly methyl acrylate and then reacted with hydrazine hydrate in presence of di-methyl sulphoxide to form poly acrylic acid hydrazide under continuous reflux and stirring then filtered and dried as shown in Figures 1 and 2.

Synthesis of modified N-Salicylidene polymer (polymerization reaction)

Poly acrylic acid hydrazide which corresponding react with Salicylaldehyde in presence of methanol and distilled water under continuous reflux and stirring then filtered and dried to obtain the new polymer prepared (modified N-Salicylidene polymer) as shown in Figure 3.

Preparation of tin complex from the reaction of N-Salicylidene derivatives of poly acrylic acid hydrazide with dimethyl tin dichloride

A solution of dimethyl tin dichloride in methanol was added drop wise with constant stirring to a suspension of

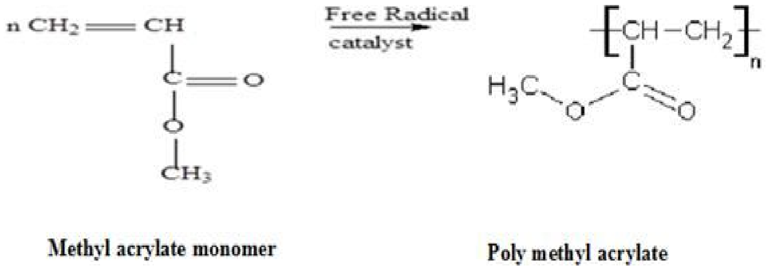
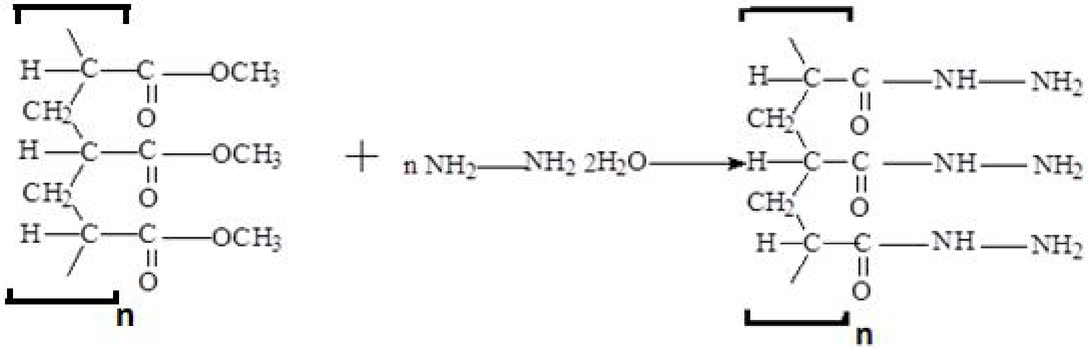


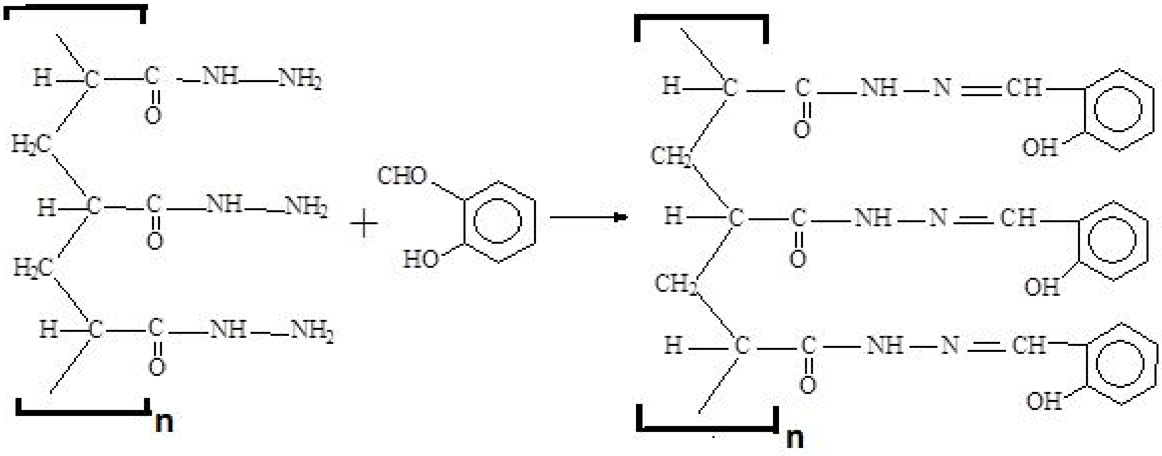
Figure 1. Structure of poly-methyl acrylate.



Poly methyl acrylate

Poly methyl acrylic acid hydrazid

Figure 2. Structure of poly methyl acrylic acid hydrazide.



Poly-methyl acrylic acid hydrazide

N-Salicylidene poly methyl acrylic acid hydrazide

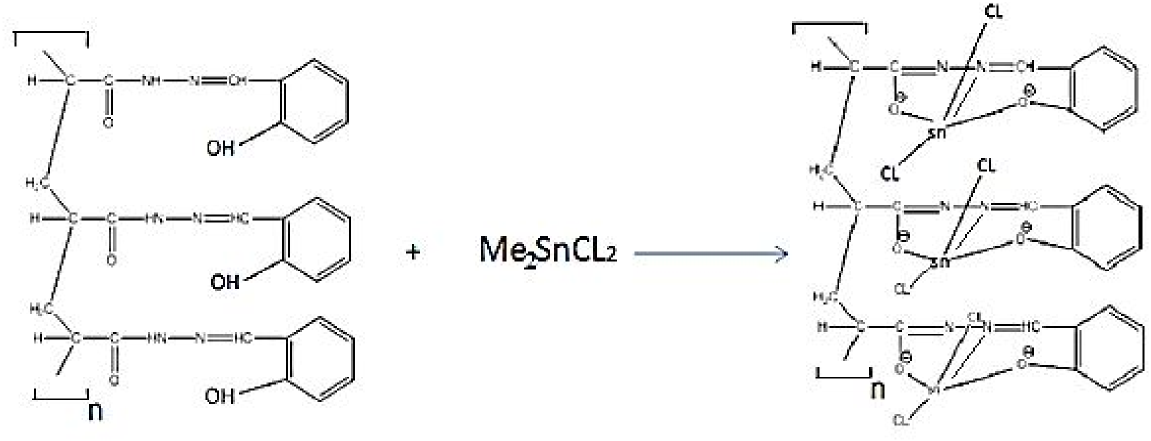
(Modified N-salicylidene polymer)

Figure 3. Structure of modified N-salicylidene polymer.

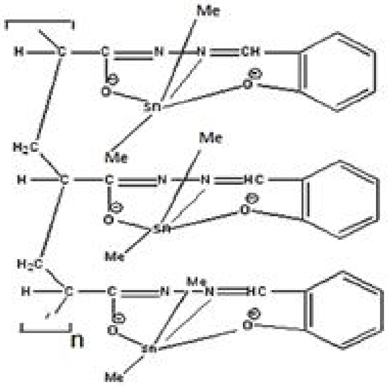
Modified N-Salicylidene Polymer in methanol, then a solution of sodium metal in methanol was added to it drop by drop. The reaction mixture was boiled under reflux with constant stirring for two hours. On cooling the resulted yellow tin complex was filtrated and washed with methanol. The new product complex is soluble in chloroform.

Pour point measurements (ASTM D-97)

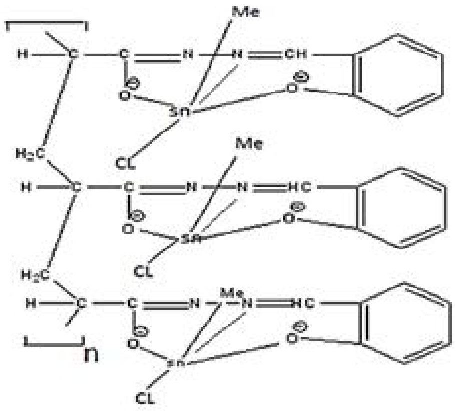
This test method is intended for use on any petroleum product, a procedure for testing the fluidity of a residual fuel oil at a specified temperature to determine the pour point of crude oil. Pour point in petroleum products is the lowest temperature at which movement of the test



Structure (a)



Structure (b)



Structure (c)

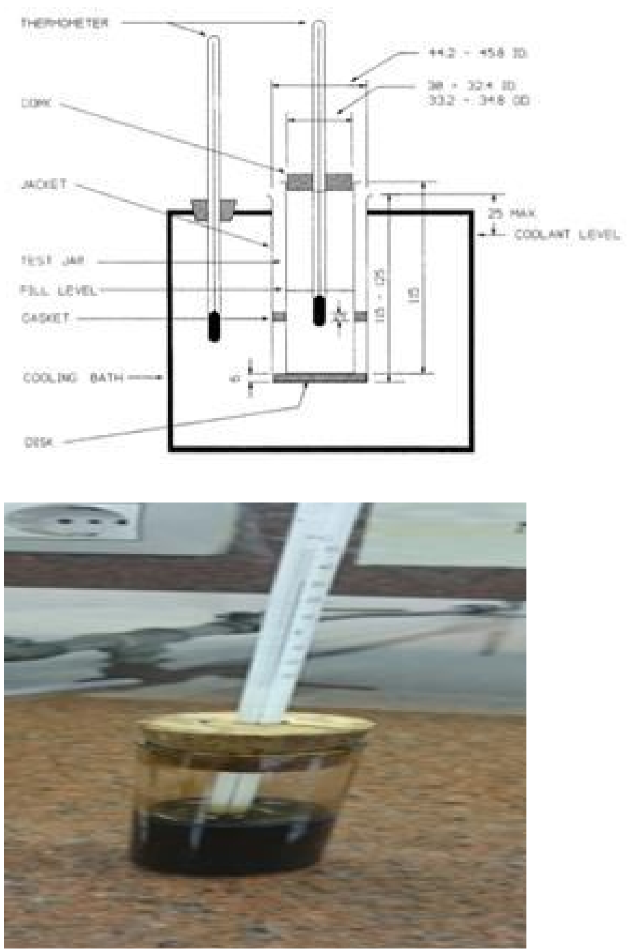


Figure 4. Pour point apparatus.

specimen is observed under prescribed conditions of test. After preliminary heating, the sample cooled at specified rate at examined at interval 3°C for flow. Characteristics the lowest temperature at which the movement of specimen is observed is recorded as the pour point as it shown in Figure 4.

Characterization methods of prepared poly-methyl acrylic acid hydrazide, modified N-Salicylidene polymer and tin chloride complex

Scanning electron microscopy (SEM)

A scanning electron microscopy (SEM) is a type of electron microscope that produces Images of a sample by scanning it with a focused beam of electrons. The electrons interact with atom in the sample producing varies signals that can be detected and that contain information about the sample's surface topography and composition.

SEM can achieve resolution better than

1 nanometer. Specimens can be observed in high vacuum, in low vacuum, and (in environmental SEM) in wet conditions. In the present work, the samples prepared were analyzed by using SEM "JEOL JSM 6360L.

FTIR spectroscopy analysis

The surface functional groups and structure were studied by FTIR spectroscopy. The FTIR spectra of the prepared samples were recorded between 500 and 4000 cm^{-1} in FTIR- 8400 S Shimadzu. FTIR spectrometer simultaneously collects high spectral resolution data over a wide spectral range. This confers a significant advantage over a dispersive spectrometer which measures intensity over a narrow range of wavelengths at a time.

Brunauer-Emmett-Teller (BET-surface area)

BET device is used in surface area measurements of powder or batch samples and pore size and pore size distribution. In the present work, the samples prepared were analyzed by using the Belsorp mini ii-Japan. The device determined needed gas quantity to cover the sample surface with a molecular layer and calculate the surface area using Brunauer, Emmett and Teller (BET) theory. Equipment analysis active surface area in m^2/g unit was carried out with chemical absorption technique. Device can be used in determination of porous structure in raw materials, in applications like moisture or gas detectors. Measureable minimum surface area is 0.05 m^2/g when nitrogen is used and 0.0005 m^2/g when cripton is used. Measureable minimum pore size is 3.5-5000 A with nitrogen and pore volume limits is less than 0.0001 cc/g. BET device, with 1°C/min heating rate, maximum 400°C can be achieved. Non-corrosive gases (N_2, Ar, CO_2, C_4H_{10} etc.), corrosive steam) NH_3 cyclo-hexane etc) can be used. For the analysis, a quantity of powder or massive sample that corresponds to 12-15 m^2 is used.

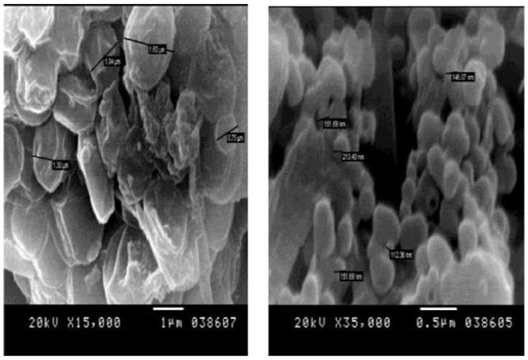
Thermo gravimetric analysis (TGA)

The thermo grams in air were obtained on a mettle TA-3000 System at a heating rate of 10 K/min at room temperature under atmospheric pressure taking 0.2 g of copolymer sample in a platinum crucible

RESULTS AND DISSCUSSION

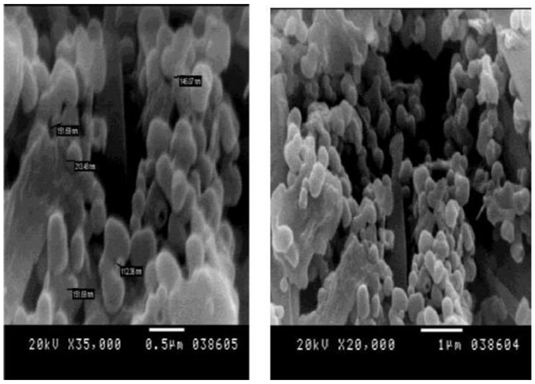
Morphology study (SEM) of prepared materials of PPD

SEM images with different levels of magnifications factor for the prepared poly acrylic acid hydrazide, modified N-salicylidene polymer and tin chloride complex as a pour point depressant with magnifications factor 35000, 15000 and 10000 respectively is shown in Figures 5a and b, 6a and b and 7a and b. These results show that polymers prepared is small spherical shape and smooth surface like agglomerate.



(a) (b)

Figure 5. a) SEM of poly-methyl acrylic acid hydrazide with magnification factor 15000. b) SEM of poly–methyl acrylic acid hydrazide with magnification factor 35000.



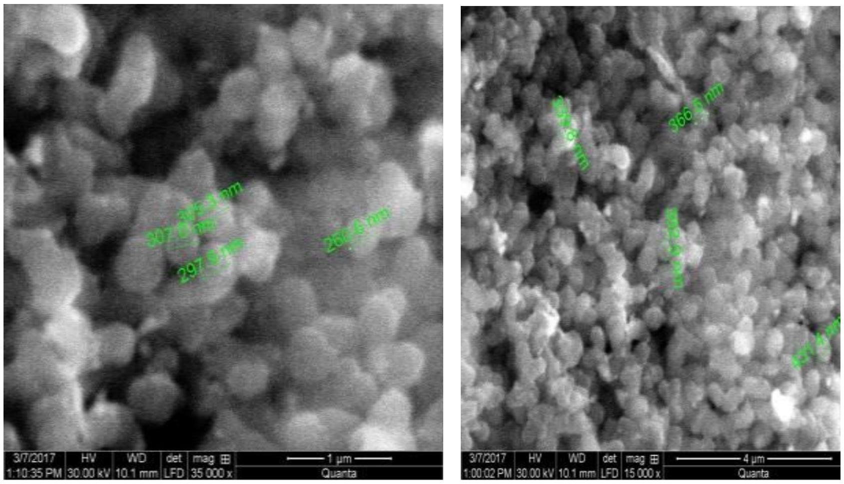
(a) (b)

Figure 6. a) SEM of modified N-salicylidene polymer with magnification factor 35000. b) SEM of modified N-Salicylidene polymer with magnification factor 20000.

Surface area

BET device is used in surface area measurements of powder or batch samples and pore size and pore size distribution as shown in Table 2.

The highest surface area for modified N-Salicylidene polymer due to the addition of high molecular weight of



(a) (b)

Figure 7. a) SEM of tin chloride complex with magnification factor 35000. b) SEM of tin chloride complex with magnification factor 15000.

Table 2. BET surface area for hydrazine, modified N-salicylidene polymer and tin chloride complex.

Filename	COMMENT	Adsorptive Adsorption temperature	$a_{s,BET}$ [m^2 g^{-1}] C
poly acrylic acid hydrazide	A_1-03-2017DAT A_1-03-2017	N2 77.000	1.7012E+01 2.0692
modified N-Salicylidene polymer	B_1-03-2017DAT B_1-03-2017	N2 77.000	7.7804E+01 3.8153
Tin chloride complex	C_1-03-2017DAT C_1-03-2017	N2 77.000	1.6892E+01 11.613

salicylaldehyde, and poly acrylic acid hydrazide also give good indication for improving the pour point.

Fourier transform infra-red (FTIR) analysis of prepared materials of PPD

IR spectra of the prepared polymers are analyzed on IR spectra using FTIR-8400 Shimadzu instrument as shown in Figures 8, 9 and 10.

Figure 8 show that the spectra of hydrazine polymer as a series of bands characteristic to the hydrazide (-CO-NH-NH_2) residue. In the 3450-3100 cm^{-1} region spectra exhibited a number of bands due to $\upsilon_{sym}(NH_2)$ symmetric stretching vibration and υ_{asym} (NH_2) asymmetric stretching vibration of the (3200 cm^{-1}) attributable to υ(NH)of the secondary NH group. Similar to amides, the hydrazide group also showed the spectra of hydrazine polymer also showed IR bands due to both C=O In plane deformation and out of plane deformation respectively at 656 cm^{-1}.

Figure 9 IR spectra of N-Salicylidene polymer showed the amide 1 bond C=O at 1623 cm^{-1} while amide 2, 3 and 4 appeared respectively at 1573, 1203 and 679 cm^{-1}. In addition to these bonds, the spectra showed strong bond at 1661 cm^{-1} due to C=N. In most cases the phenolic group [OH] of the N-Salicylidene derivatives cannot be identified due to the strong inter hydrogen bonding with C=N.

The IR spectra of tin chloride complex polymer did not lack any absorption due to NH_2. The IR Spectrum of tin complex polymer shows NH bond at 3000 cm^{-1} beside a strong bond at 1615 cm^{-1} attributable to coordination C=O amide 1 bond. The amide 2 and 3 and amide 4 appear at 1573, 1201 and 656 cm^{-1}, respectively.

Comparison of IR spectra of tin complex with the uncoordinated N-Salicylidene polymer reveal that the amide 1 bond is shifted towards lower frequencies 8 cm^{-1} due to coordination of the hydrazide carbonyl oxygen due to the central of ion metal.

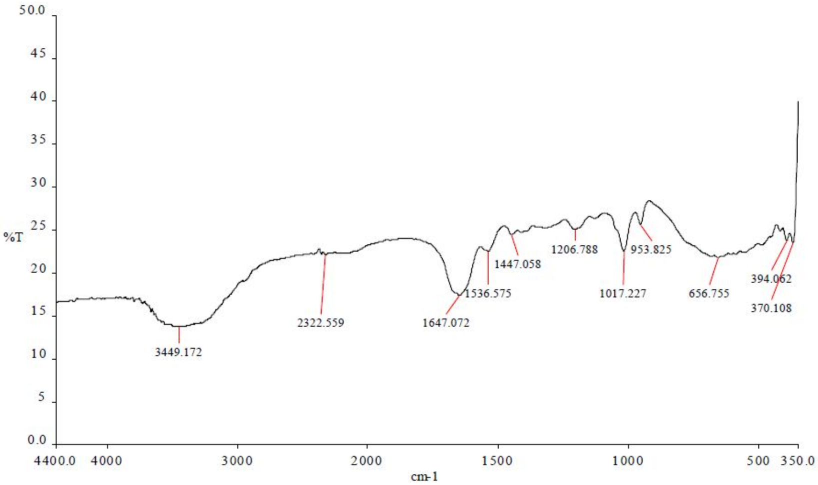


Figure 8. FTIR of poly-methyl acrylic acid hydrazide.

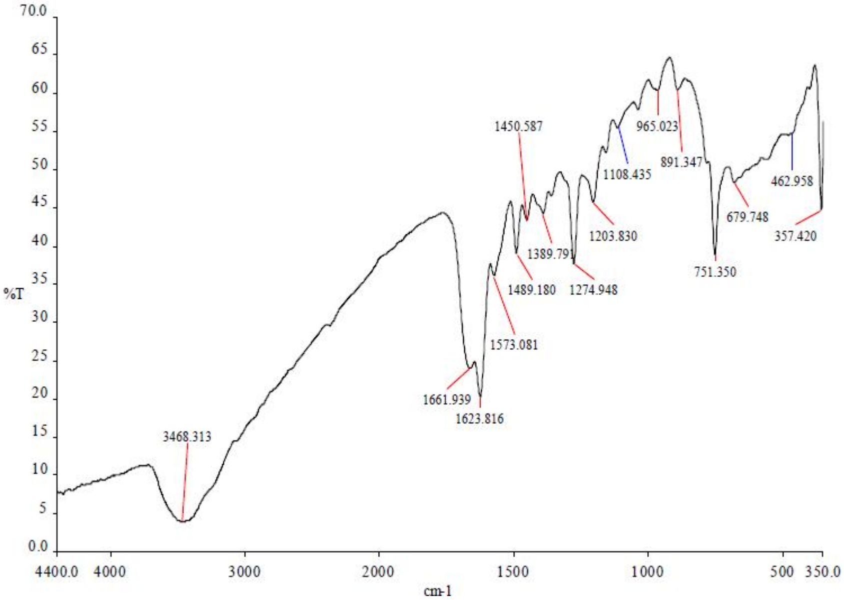


Figure 9. FTIR of modified N-salicylidene polymer.

Thermo gravimetric analysis of prepared materials as PPD

TGA curves of poly acrylic acide hydrazide in Figure 11 shows many stages where stage 1 occurs in temperature range (61 to 117.7°C) due to small loss of H_2O (entrained water) 3.36% loss. Stage 2 and stage 3 occur in temperature range (117.7 to 239.9°C) due to continuous rapture and releasing $NH\text{-}NH_2$ in the whole molecules which represent about 37.2%. Stage 4 occurs in temperature range (239.9 to 323.8°C) due to rapture of C=O bond in the whole molecule. Stage 5 occurs in temperature range (323.8 to 445.9°C) due to rapture between [C-C] bond in the molecule. The last two stages occur in the temperature range (445.9 to 598.8°C)

Modified N-Salicylidene polymer as shown in Figure 12, stage 1 occurs at temperature range (32.7 to 162.8°C) due to small loss of H_2O molecules (entrained water) 7.1% loss, while stage 2 and stage 3 occur in temperature range (162.8 to 369.79°C) due to the continuous double bond rapture between N=CH in the whole molecule which represent about 54% loss, the last stage occur in temperature range (369.7 to 598.2°C) due to further molecule degradation.

Figure 13 shows the TGA of tin chloride complex, when stage 1 occurred at temperature range (58.5 to 191.1°C) due to loss of H_2O molecule (entrained water 3.5%).

Stage 2 occurs in the temperature range (191.7 to

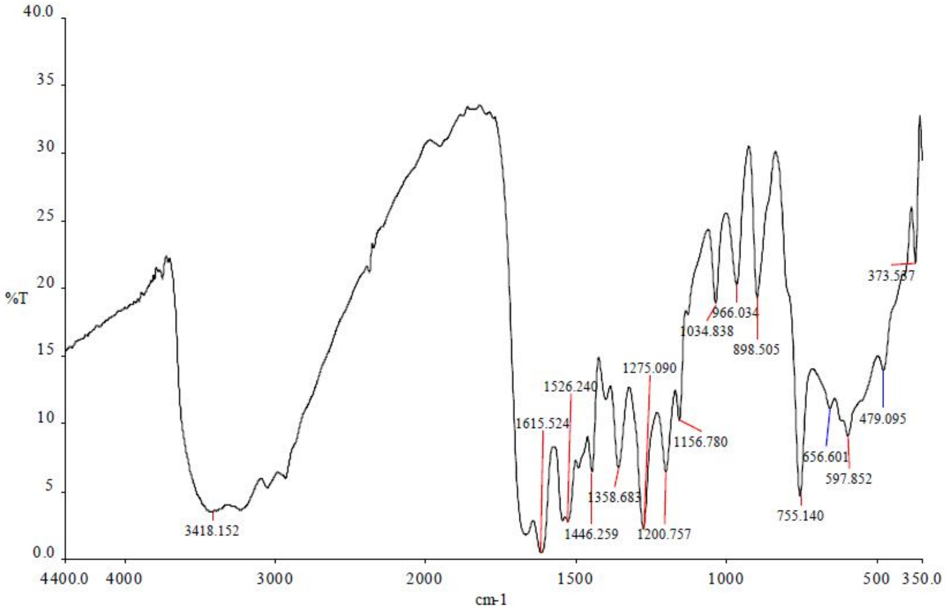
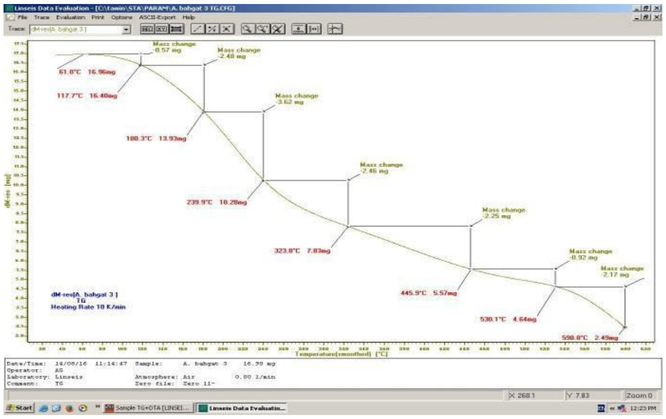


Figure 10. FTIR of tin chloride complex.



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Date/Time:   14/08/16  11:14:47  Sample:      A. bahgat 3    16.90 mg
Operator:    AG
Laboratory:  Linseis             Atmosphere:  Air            0.00 l/min
Comment:     TG                  Zero file:   Zero 11-
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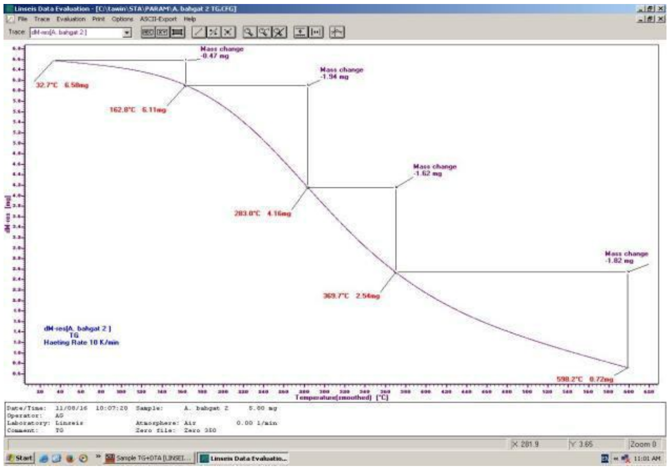
Figure 11. TGA of poly-methyl acrylic acide hydrazide.



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Date/Time:   11/08/16  10:07:20  Sample:      A. bahgat 2    5.60 mg
Operator:    AG
Laboratory:  Linseis             Atmosphere:  Air            0.00 l/min
Comment:     TG                  Zero file:   Zero 350
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Figure 12. TGA of modified N-salicylidene polymer.

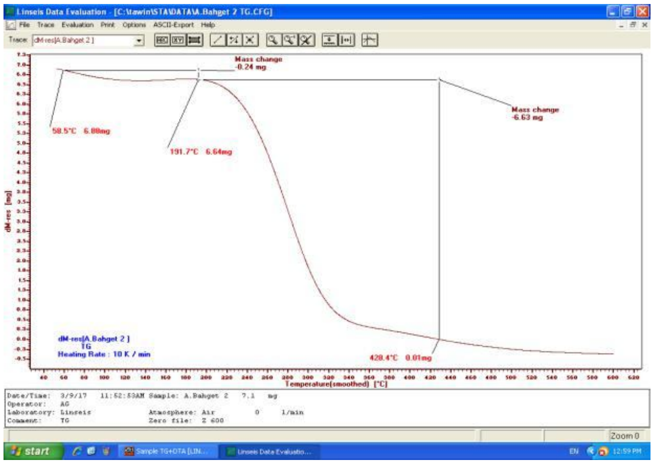
420.4°C) due to dissociation of metal complex formed.

Comparison of effect between different types of poly acrylic acid hydrazide, modified N-Salicylidene and tin chloride complex on Alamin crude oil

Figure 14 shows a comparison among different types of pour point depressants poly acrylic acid hydrazide, modified N-Salicylidene and tin chloride complex concentration (ppm) on Alamin crude oil. It is obviously shows that the pour point stay constant at 6°C as poly acrylic acid hydrazide concentration increase till 300 ppm, while modified N-salicylidene and tin chloride complex both drop the pour point to 3°C at 300 ppm. In addition, the three depressant have the same performance at 400 ppm and pour point become 3°C and then dramatically dropped from 3 to 0°C at 500 ppm. The results obtained that poly acrylic acid hydrazide, modified N-saliclidene and tin chloride complex make the best performance at 500 ppm.

Comparison of effect between different types of poly acrylic acid hydrazide, modified N-Salicylidene and tin chloride complex on Qarun crude oil

Figure 15 shows a comparison among different types of pour point depressants poly acrylic acid hydrazide, modified N-Salicylidene and tin chloride complex concentration (ppm) on Qarun crude oil. It obviously shows that the pour point stay constant at 27°C as poly acrylic acid hydrazide, modified N-salicylidene and tin chloride complex concentration increase at 100 ppm,



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Date/Time:   3/9/17   11:52:53AM Sample: A.Bahget 2   7.1   mg
Operator:    AG
Laboratory:  Linseis            Atmosphere: Air        0     1/min
Comment:     TG                 Zero file:  Z 600
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Figure 13. TGA of tin chloride complex crystal.

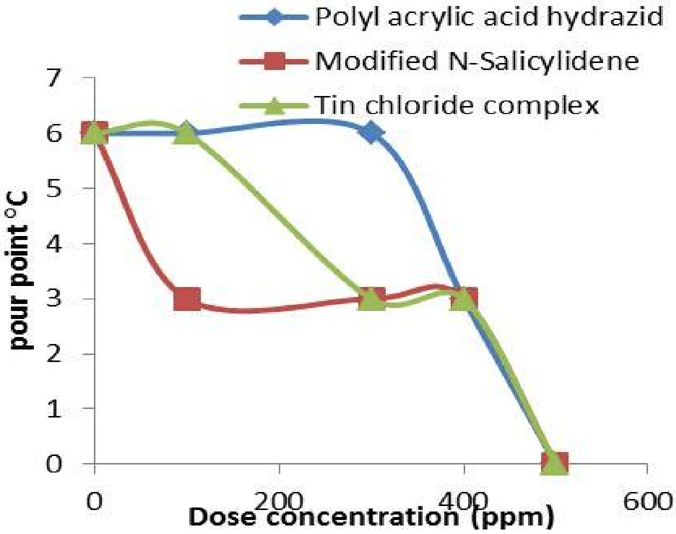

Figure 14. Comparison between three pour point depressant on Alamin crude oil.

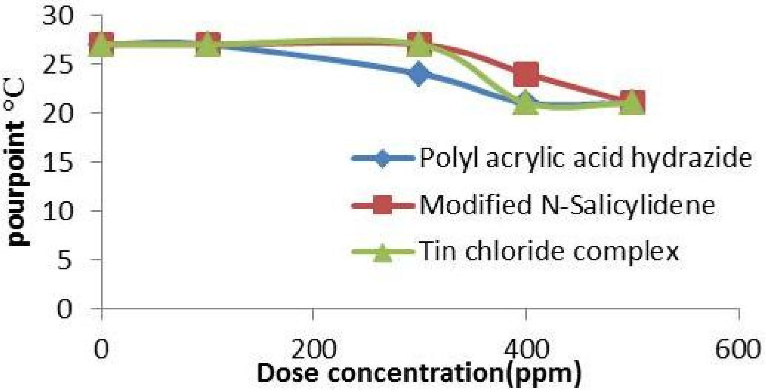

Figure 15. Comparison between three pour point depressant on Qarun crude oil.

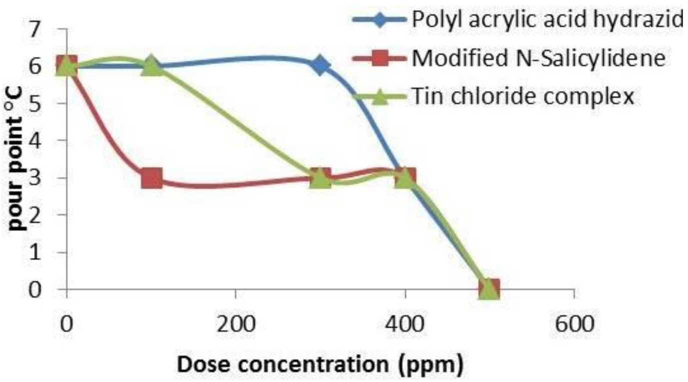

Figure 16. Comparison between three pour point depressant on Alamin crude oil after two hours.

while in case of poly acrylic acid hydrazide the pour point decreased to 24°C at 300 ppm. In addition, the increasing concentration of poly acrylic acid hydrazide and tin chloride complex to 400 ppm reduce the pour point to 21°C. The pour point becomes constant at 500 ppm and reach to 21°C for the three depressants. It obtained from the results that modified N-Salicylidene and tin chloride complex is recommended for Qarun crude oil at 400 ppm.

Comparison of effect between different types of poly acrylic acid hydrazide, modified N-salicylidene and tin chloride complex on Alamin crude oil after two hours

Figure 16 shows a comparison among different types of pour point depressants poly acrylic acid hydrazide, modified N-Salicylidene and tin chloride complex concentration (ppm) on Alamin crude oil after two hours. It obviously shows that the pour point stays constant at 6°C as poly acrylic acid hydrazide concentration increase till 300 ppm, while modified N-salicylidene and tin chloride complex both drop the pour point to 3°C at 300 ppm. In addition, the three depressant have the same performance at 400 ppm and pour point become 3°C and then dramatically dropped from 3 to 0°C at 500 ppm .the results obtained that poly acrylic acid hydrazide, modified N-salicylidene and tin chloride complex make the best performance at 500 ppm. This result is in agreement with the literature of Borthakur et al. (1996).

Comparison of effect between different types of poly acrylic acid hydrazide, modified N-salicylidene and tin chloride complex on Qarun crude oil after two hours

Figure 17 shows a comparison among different types of pour point depressants poly acrylic acid hydrazide, modified N-salicylidene and tin chloride complex (ppm) on Qarun crude oil after two hours. It obviously shows that the pour point stay constant at 27°C as poly acrylic acid hydrazide, modified N-salicylidene and tin chloride complex concentration increase at 100 ppm, while in case of poly acrylic acid hydrazide the pour point decreased to 24°C at 300 ppm. In addition, the increasing concentration of poly acrylic acid hydrazide and tin chloride complex to 400 ppm reduce the pour point to 21°C. The pour point becomes constant at 500 ppm and reaches 21°C for the three depressants. It is obtained from the results that modified N-salicylidene and tin chloride complex are recommended for Qarun crude oil at 400 ppm. The result obtained after two hours is that there is no effect of time on the performance of poly acrylic acid hydrazide, modified N-salicylidene and tin chloride complex.

Comparison of effect between different types of poly acrylic acid hydrazide, modified N-salicylidene polymer and tin chloride complex on Alamin crude oil after 24 hours

Figure 18 shows a comparison among different types of pour point depressants poly acrylic acid hydrazide, modified N-salicylidene and tin chloride complex concentration (ppm) on Alamin crude oil after 24 h. It is obviously shows that the pour point stay constant at 6°C as poly acrylic acid hydrazide and tin chloride complex concentration increase at 100 ppm, while modified N-salicylidene drops the pour point to 3°C at 100 ppm. In addition, the three depressants have the same performance at 300 ppm and pour point become 3°C and then pour point dramatically dropped from 3 to 0°C at 400 ppm for poly acrylic acid hydrazide and modified N-salicylidene, while pour point in case of tin chloride complex stayes constant at 3°C when the concentration increase to 400 ppm. The results obtained that poly acrylic acid hydrazide and modified N-salicylidene gives the best performance at 400 ppm of Alamin crude oil.

Comparison of effect between different types of poly-acrylic acid hydrazide, N-salicylidene and tin chloride complex on Qarun crude oil after 24 hours

Figure 19 shows a comparison among different types of pour point depressants poly-acrylic acid hydrazide, modified N-salicylidene and tin chloride complex concentration (ppm) on Qarun crude oil after 24 h. It obviously shows that the pour point stay constant at 27°C as poly acrylic acid hydrazide, modified N-salicylidene and tin chloride complex concentration increase at 100 ppm, while in case of poly-acrylic acid hydrazide the pour point decreased to 21°C at 300 ppm. In addition, the increasing concentration of modified N-salicylidene polymer to 300 ppm reduces the pour point to 24°C. The pour point becomes constant at 400 ppm and reached

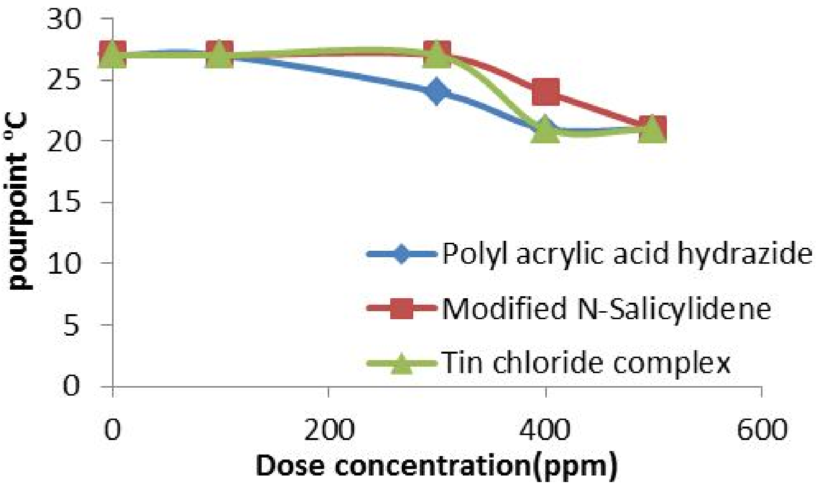

Figure 17. Comparison between three pour point depressants on Qarun crude oil after two hours.

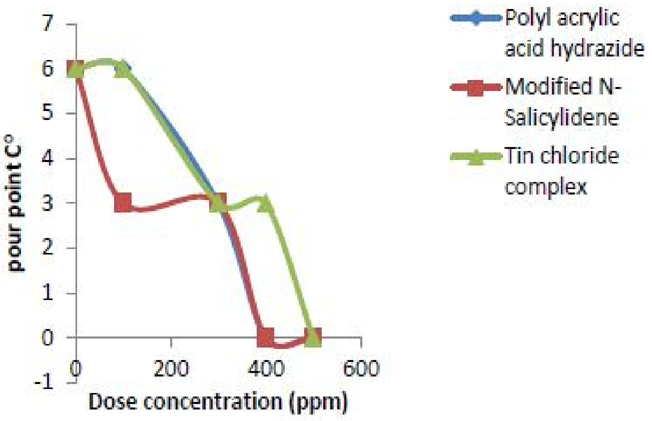

Figure 18. Comparison between three pour point depressant on Alamin crude oil after 24 hours.

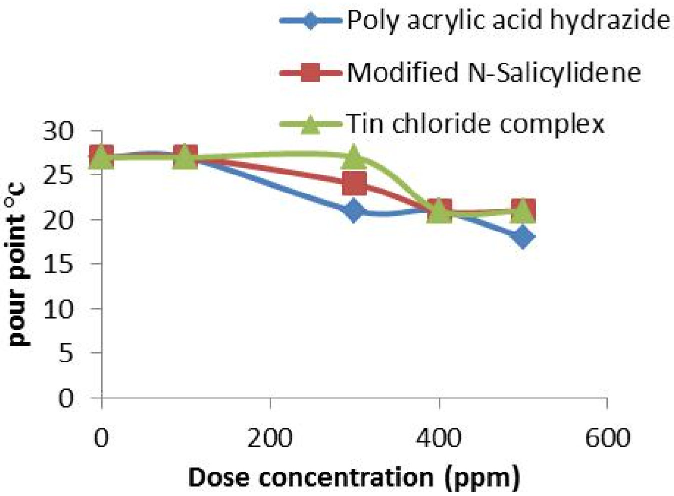

Figure 19. Comparison between three pour point depressant on Qarun crude oil after 24 hours.

21°C for the three depressants. on the other hand, the pour point decreased sharply to 18°C at 500 ppm in case of using poly acrylic acid hydrazide.it obtained from the results that poly acrylic acid hydrazide has best performance on Qarun oil at 500 ppm .

Comparison between modified N-salicylidene polymer prepared and different other pour point depressant (PPD) additives

As shown in Table 3, there are many PPD that can be used for different types of waxy crude oil. The modified N-salicylidene polymer prepared shows similar performance as the PPD in the literature.

CONCLUSIONS

A new materials had been prepared that are effective for improving the flow of both types of Egyptain waxy crude oil, different polymeric additives were synthesized and characterized in the laboratory and their effects on pour point were investigated. All the additives satisfy most of the requirements to act as a pour point depressant and flow improvers. Based on the results of this study, the conclusion can be drawn in the following:

Modified N-salicylidene polymer has the highest surface area for the three prepared materials that works as pour point depressant on two different type of Egyptian waxy crude oil Alamin and Qarun.

The tin chloride complex has the most stable compound for temperature degradation which indicate low surface area corresponding to the other materials prepared that confirm the presence in a complex form.

The three PPD prepared show good results for Alamin and Qarun crude oil at 500 ppm that decrease the pour

Table 3. Comparison between prepared flow improvers and other PPD additives.

Crude type	Concentration in ppm					PPD used	Reference no.
	Pour point °C						
	Nil	100	300	400	500		
Alamin(Egypt)	6	6	6	3	0	Poly-methyl acrylic acide hydrazide	Current work
Qarun (Egypt)	27	27	24	21	21	Poly-methyl acrylic acide hydrazide	Current work
Alamin(Egypt)	6	6	3	3	0	Tin chloride crystal	Current work
Qarun (Egypt)	27	27	27	21	21	Tin chloride crystal	Current work
Alamin(Egypt)	6	3	3	3	0	Modified N-Salicylidene polymer	Current work
Qarun (Egypt)	27	27	24	21	21	Modified N-Salicylidene polymer	Current work
BH (Indian)	30	--	12	9	9	Alkyl Fumarate-Vinyl Acetate Copolymer	Borthakur et al. (1996)
MRN (Indian)	30	--	15	12	9	Alkyl Fumarate-Vinyl Acetate Copolymer	Borthakur et al. (1996)
GLK (Indian)	33	--	15	12	12	Alkyl Fumarate-Vinyl Acetate Copolymer	Borthakur et al. (1996)
Khalda (Egypt)	14	11	14	14	14	NAFOL2022methaacrylate homopolymer	El-Gamal et al. (1994)
Salam(Egypt)	21	9	3	3	0	NAFOL2022methaacrylate homopolymer	El-Gamal et al. (1994)
Khalda (Egypt)	14	14	14	14	14	NAFOL1822methaacrylate homopolymer	El-Gamal et al. (1994)
Salam(Egypt)	21	6	3	3	0	NAFOL1822methaacrylate homopolymer	El-Gamal et al. (1994)
Khalda (Egypt)	14	5	11	14	14	NAFOL2022acrylate- NAFOL2022 metha acrylate copolymer	El-Gamal et al. (1994)
Salam(Egypt)	21	18	9	3	3	NAFOL2022acrylate- NAFOL2022 metha acrylate copolymer	El-Gamal et al. (1994)
Khalda (Egypt)	14	-1	5	5	8	NAFOL2022acrylate-vinyl acetate copolymer	El-Gamal et al. (1994)
Salam(Egypt)	21	15	0	0	0	NAFOL2022acrylate-vinyl acetate copolymer	El-Gamal et al. (1994)

point from 6 to 0°C and from 27 to 21°C, respectively. The three PPD prepared are very stable with time in high performance as staying overnight means the additive is stable with time.

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