

Synthesis and Characterization of Polymyristyl Acrylate as a Potential Additive for Lubricating Oil

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Abstract Homopolymer of myristyl acrylate was synthesized by two different ways; microwave (MW) assisted method and thermal method using benzoyl peroxide (BZP) as initiator. The polymers were characterized by spectroscopic (NMR, IR) and viscometric analysis. Additive performances of each of them [pour point depressant (PPD) and viscosity modifier (VM)] for lubricating oil (lube oil) were also evaluated by standard ASTM methods. Oil thickening property of the polymer has also been investigated and reported here. Thermal stability of the polymer was determined by thermo gravimetric analysis. A comparison of their performances has also been reported. The MW assisted method has appeared to be a cost effective greener approach.

Keywords Polymyristyl Acrylate, PPD, Viscosity Index Improver, ASTM Method

1. Introduction

Modern lubricants are formulated from a range of base fluids and chemical additives. The base fluids has several functions but primarily it is the lubricant, providing a fluid layer separating moving surfaces or removing heat and wear particles while keeping friction at a minimum. Many of the properties of the lubricant are enhanced or created by the addition of special chemical additives to the base fluid. For example viscosity modifier (VM) or viscosity index improver (VII) [1], pour point depressant (PPD)[2], antioxidant[3], corrosion inhibitor[4], extreme pressure agent[5] etc. The base fluid also functions as the carrier for these additives in solution under all normal working conditions. Multifunctional additives satisfy more than one purpose and hence research throughout the world is increasing directed toward producing such type additives. In order to improve the pour point (the lowest temperature at which the oil stays flowing) and to meet the requirements of the multigrade engine oil specifications pour point depressants (PPD) are added. PPD functions by depositing or adsorbing on the wax crystals network by markedly reducing the size of the wax crystals formed. Viscosity index improvers(VII) also known as viscosity modifier(VM) are long chain, high molecular weight polymers used to resist the change of viscosity of the oil by increasing the relative viscosity of oil more at high temperatures than at low temperatures[6-9]. The performance of VMs is very often expressed in terms of Viscosity Index

(VI), which is an arbitrary number[10] that indicates the resistance of a lubricant to viscosity change with temperature. The higher the VI, the less the viscosity of an oil changes for a given temperature change. The performance of the VII depends on the behavior of the polymer molecules in the oil, where the polymer solubility, molecular weight and resistant to shear degradation are determinant parameters[9]. Oil thickening property of the polymer, which is a direct measure of percent increase in the viscosity of the base stocks for addition of its unit amount of weight, has also been investigated and reported here. This property can also be taken as the measure of extent of interaction of the polymer with the base stock, greater the thickening property; greater is the extent of interaction[11]. Fuel economy may also be predicted by the thickening power of a lube oil additive. Although additives of many diverse types have been developed to meet special lubrication needs, acrylate based polymers as lube oil additive are widely used. Several kinds of poly alkyl acrylates are generally used as performance additive[12] especially as PPD and VM in lubricating oil composition. In this paper the additive properties of polymyristyl acrylate have been investigated.

Microwave irradiation is a well-known method for heating and drying materials and is utilized in many private households and industrial applications for this purpose. Both organic and inorganic reactions undergo an immense increase in reaction speed under microwave irradiation compared with conventional heating.[13] Significant improvements in yield and selectivity have also been observed as a consequence of the fast and direct heating of the reactants themselves. Furthermore, high-pressure synthesis is easily accessible for reactions performed in closed vessels, facilitating the use of low boiling solvents and thereby pav-

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ing the way to environmentally benign reaction conditions.

With this view in mind and as a part of our ongoing studies on the synthesis and evaluation of polymeric additives, we have undertaken the synthesis of the polymyristyl acrylate under thermal as well as microwave condition. The additives have been prepared, characterized and their performances as PPD and viscosity modifier (VM) along with the thickening properties in different base oils and have been evaluated and compared.

Physical characterization of the polymers was carried out employing spectroscopic (IR and NMR) techniques and thermo gravimetric analysis (TGA). The behavior of the polymeric additives towards a specific solvent / base stock plays a significant role in their action as a performance additive in their end application. Viscometry provides very important data about the interaction of additive in base fluid and hence conformation of polymeric system in the base stock. Again, reports regarding such information are scanty [14] and almost nil for polymers used as lube oil additives, present research also include viscometric study of this polymer. In addition, because of the simplicity of the procedure, viscometry is usually employed to complement the results obtained from other techniques, generally in determination of molecular mass of samples with the available literature value of the constants used in the particular equation.

The relationship between viscometric molecular weight and intrinsic viscosity $[\eta]$ can be described by the Mark-Houwink-Sakurada (MHS) equation:

$$[\eta] = KM^a \quad (1)$$

Where, $[\eta]$ is the intrinsic viscosity in dLg^{-1} , M is the viscosity-average molecular weight, and K and a are viscometric constants for given solute-solvent system and temperature. The higher the molecular weight, the more viscous the polymer solution will be. This is reasonable, when a polymer has a higher molecular weight, it has a bigger hydrodynamic volume; that is, the volume that the coiled up polymer takes up in solution. Being bigger, the polymer molecule can block more motion of the solvent molecules. It might be said that it can block off more lanes of the highway. Also, the bigger a polymer is, the stronger its secondary forces are. So the higher the molecular weight, the more strongly the solvent molecules will be bound to the polymer. This enhances the slowing-down of the solvent molecules. A number of mathematical relations are available in literature for the study of viscometric properties of a dilute polymer solution at a particular temperature. The most commonly used equations are:-

$$\text{Huggins(H)} \quad \eta_{sp}/C = [\eta]_h + k_h [\eta]_h^2 C \quad (2)$$

$$\text{Kraemer(K)} \quad \ln \eta_r / C = [\eta]_k - k_k [\eta]_k^2 C \quad (3)$$

$$\text{Martin(M)} \quad \ln(\eta_{sp}/C) = \ln[\eta]_m + k_m [\eta]_m C \quad (4)$$

$$\text{Schulz-Blaschke(SB)} \quad \eta_{sp}/C = [\eta]_{sb} + k_{sb} [\eta]_{sb} \eta_{sp} \quad (5)$$

$$\text{Solomon-Ciuta(SC)} \quad [\eta] = [2(\eta_{sp} - \ln \eta_r)]^{1/2} / C \quad (6)$$

$$\text{Deb-Chatterjee(DC)} \quad [\eta] = (3 \ln \eta_r + 3/2 \eta_{sp}^2 - 3 \eta_{sp})^{1/3} / C \quad (7)$$

Where, $\eta_r = t/t_0$ = time of flow of the solution / time of flow of the pure solvent = relative viscosity or viscosity ratio
 $\eta_{sp} = \eta_r - 1$ = specific viscosity

$[\eta]_h$ = intrinsic viscosity, respective to Huggins equation

$[\eta]_k$ = intrinsic viscosity, respective to Kraemer equation

$[\eta]_m$ = intrinsic viscosity, respective to Martin equation

$[\eta]_{sb}$ = intrinsic viscosity or limiting number, respective to Schulz-Blaschke equation.

kh, kk, km and ksb are Huggins, Kraemer, Martin and Schulz-Blaschke coefficients, respectively.

The equations 2 to 5 are used for the determination of intrinsic viscosity by graphical extrapolation method [15-19]. The last two (eqs. (5),(6)) are applied for single point determination [16-18]. This method has the advantage of being considerably faster and can be adequate when a large number of samples must be analyzed in short period of time, practically in industrial laboratories.

The use of these equations have been derived under the supposition of the validity of the relationship $kh + kk = 0.5$ [16]. Schultz-Blaschke equation is also applied for single point determination where the value of $K_{sb} = 0.28$. Your goal is to adhere to this paper in appearance as closely as possible.

2. Experimental Part

2.1. Materials

Myristyl alcohol ($\text{CH}_3-(\text{CH}_2)_{13}-\text{OH}$, 98%, SRL India), and acrylic acid ($\text{H}_2\text{C} = \text{CH}-\text{COOH}$, 99%, Merck) were taken without further purification. Benzoyl peroxide (BZP) ($(\text{C}_6\text{H}_5\text{CO})_2\text{O}_2$, 98 %, LOBA chemie, India) was used after recrystallization from CHCl_3 -MeOH mixture. Toluene was purified by distillation after being refluxed for 2 h in presence of sodium and used as a solvent for esterification. Concentrated sulfuric acid (98%, Merck) as a catalyst and hydroquinone (99%, Merck) as an inhibitor were also used for esterification. Base oils were collected from IOCL, India and BPCL, India.

2.2. Procedures

2.2.1. Preparation and Purification of the Ester of Myristyl Alcohol

The ester was prepared by reacting 0.5 mole of acrylic acid with 0.5 mole myristyl alcohol. The reaction was carried out in a resin kettle in the presence of concentrated sulphuric acid as a catalyst, 0.25% hydroquinone as polymerization inhibitor for acrylic acid and toluene as a solvent. The esterification reaction was carried out under a slow stream of deoxygenated nitrogen. The reactants, which were mixed with toluene, were heated gradually from room temperature

to 403 K using a well-controlled thermostat. The extent of reaction was followed by monitoring the amount of liberated water to give the ester.

2.2.2. Purification of the Prepared Acrylate Ester

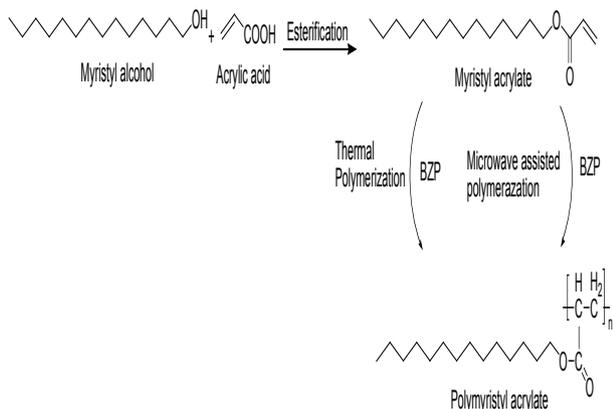
The prepared ester was purified by the method as described in our earlier publication[20].

2.2.3. Thermal Polymerization

Polymerization was carried out by following the procedure as reported elsewhere[21].

2.2.4. Microwave Assisted Polymerization

Polymerization was carried out in a focused mono-mode microwave oven(CEM corporation, Matthews, NC) applying 300 WT for 15 minute at 90°C without any solvent by adding 0.01% (w/w) BZP as initiator[22].



Scheme 1. Polymerization of Myristyl acrylate Prepared from Myristyl Alcohol and Acrylic Acid by Esterification in Two Different Ways

3. Measurements

3.1. Spectroscopic Measurements

Spectroscopic IR spectra were recorded on a Shimadzu FT-IR 8300 spectrometer using 0.1 mm KBr cells at room temperature within the wave number range 400 to 4000 cm^{-1} . NMR spectra were recorded in Bruker Avance 300 MHz FT-NMR spectrometer using 5 mm BBO probe. CDCl_3 was used as solvent and TMS as reference material.

3.2. Viscometric Measurements

Viscometric properties were determined at 313 K in toluene solvent following the method reported elsewhere [20].

3.3. Thermo Gravimetric Analysis (TGA)

The thermo grams in air were obtained on a mettler TA – 3000 system, at a heating rate of 10°C / min.

3.4. Evaluation of Thickening Property

Kinematic viscosity of the base oils and that of the polymer doped base oils in different concentrations were evalu-

ated. Thickening power of the polymer was determined by evaluating the percent increase in viscosity of the base stocks by the addition of unit amount of additive.

3.5. Evaluation of Pour Point

The prepared additives were evaluated as pour point depressant using base oils(BO1,BO2,BO3), each of them collected from two different sources(S1,S2), through the pour point test according to the ASTM-D97 method using WIL-471 cloud and pour point test apparatus model 3(India). The effect of additive concentration was investigated by using different doping concentrations. The experimental data were noted by taking an average of three experimental results under identical conditions.

3.6. Evaluation of Viscosity Index

Viscosity index of the prepared homopolymers were evaluated in different base stocks according to ASTM D 2270-87 method. The kinematic viscosities of the oils contain the tested compounds which were determined at 40°C and 100°C. Different concentrations ranging from 1.0 to 5.0 wt% were used to study the effect of additive concentration on(VI).

4. Results and Discussion

The IR absorption at 1732.0 cm^{-1} (P-1), 1732.9 cm^{-1} (P-2) showed the presence of ester carbonyl group of thermally prepared additive and that of prepared by microwave method (P-2) respectively. The broad peaks in the range 1259.4 cm^{-1} and 1263.3 cm^{-1} due to the ester C-O stretching vibration for P-1 and P-2 respectively. Broad peak in the range 1170.7 cm^{-1} to 1056.9 cm^{-1} for C-H stretching vibrations for both the polymers. Peaks in the range 984.6 cm^{-1} to 668.3 cm^{-1} was due to C-H bending vibrations. The ^1H NMR signal at 3.641 and 3.642 ppm indicated the presence of $-\text{OCH}_2$ group of acrylate chain. Peaks in the range 1.613 to 0.858 ppm were due to C-H bonds of the alkyl groups. Absence of peak between 5 and 6 ppm for P-1 to P-2 polymers indicated the absence of olefinic double bond which indicates effective polymerization in both the case.

The TGA data showed(Table 1) that the thermal stability of P-2 is slightly better than P-1. From this result it may also be said that the P-2 is more linear than P-1 which is quite expected.

The intrinsic viscosity values for both the polymers, obtained from different equations(eq. 1 to eq. 6), have little difference(Table 2). The higher values indicate that both the polymers have fairly strong interaction with the solvent. k_h values also indicate good solvation which is further supported by the $k_h + k_k$ values(Table 3), and thus point towards the formation of the chain like structure of the present polymer as discussed earlier[20]. Molecular weights (viscosity average) for P-1 and P-2 obtained by Mark-Houwink equation using intrinsic viscosity obtained from different equations(eq. 1 to eq. 6) showed excellent correlation(Table

4).

Table 1. Thermal Stability of the homopolymer (TGA data) *Decom. Temp.* Decomposition temperature, *PWL* Percent Weight Loss, P-1 thermally prepared polymer P-2 microwave induced polymer

Sample	Decom. Temp., °C	PWL
P-1	250/360	12/82
P-2	277/395	14/91

Table 2. Intrinsic viscosity values a- viscometric method, b- single point determination method h, k, m, sb, sc and dc refers Huggins, Kraemer, Martin, Schulz- Blaschke, Solomon-Ciuta and Deb-Chatterjee equations respectively

Sample	$[\eta]^a_h$	$[\eta]^a_k$	$[\eta]^a_m$	$[\eta]^a_{sb}$	$[\eta]^b_{sb}$	$[\eta]^b_{sc}$	$[\eta]^b_{dc}$
P-1	5.397	5.547	5.748	5.873	5.718	5.733	6.097
P-2	5.565	5.717	5.936	6.2	5.878	5.9	6.288

Table 3. Viscometric constant values obtained for the two polymeric samples. k_h , k_k , k_m and k_{sb} are Huggins, Kraemer, Martin and Schulz- Blaschke coefficients respectively

Sample	k_h	k_k	k_m	k_{sb}	$k_h + k_k$
P-1	0.494	0.108	0.316	0.23	0.602
P-2	0.497	0.106	0.316	0.228	0.603

Table 4. Molecular weight values (g/mole) determined by Mark-Houwink equation $[\eta] = KMa$ where, $K = 0.00387$ and $a = 0.725$. h, k, m, sb, sc, dc refer to the Huggins, Kraemer, Martin, Schulz- Blaschke, Solomon- Ciuta and Deb -Chatterjee respectively. a- extrapolation of graph, b- single point determination

Sample	M_h^a	M_k^a	M_m^a	M_{sb}^a	M_{sb}^b	M_{sc}^b	M_{dc}^b
P-1	21735	22573	23709	24423	23538	23623	25717
P-2	22674	23532	24785	26318	24451	24578	26834

Thickening power of both the homopolymers (Table 6) as evaluated in different base stocks (Table 5) indicated a gradual decrease with the increase in concentration of the additive. This may be because of the fact that the polymer molecule assumes a coiled like aggregation with increase in its concentration in the base stock. The result also showed that the thickening power of the P-1 polymer was slightly higher in comparison to the P-2 polymer. This indicates that as far as the fuel economy is concerned the thermal method is better than the microwave method.

The prepared homopolymers were tested for their effectiveness as viscosity index improvers by measuring the VI of the respective additive doped base oils and the values are given in Table 7. The test method (ASTM D 2270-87 method) comprises the determination of the kinematic viscosities of the different base oils containing different concentrations of the additives (ranging between 1% to 5%) at 40^o and 100^o C[23]. The data indicated that the VI increases with increasing the concentration of the prepared additives in solution and the VI values of the P-2 polymer are slightly higher in compared to the P-1 polymer in BO1 and BO2 base oils(Figure 1 & 2), whereas in BO3 base oil the values are

nearly equal for both the polymers(Figure 3). As the temperature is raised, the lube oil viscosity decreases; meanwhile the polymer molecule expands and as a result of which its hydrodynamic volume increases. This increase in the micelle size of the solvated polymer molecules counterbalance the reduction of the viscosity of the lube oils with temperature[24]. This effect is more pronounced in case of P-2 polymer rather than P-1. The increase in concentration of polymer leads to an increase in the total volume of the polymer micelles in the oil solutions. Consequently, higher concentration of additive will impart higher viscosity index [25].

Table 5. Base oil properties

Properties	BO1	BO2	BO3
Density (g.cm ⁻³) at 40°C	0.84	0.94	0.97
Viscosity at 40°C in cSt	6.7	24.229	110.053
Viscosity at 100°C in cSt	1.77	4.016	10.526
Viscosity Index	80.05	89.02	82.01
Cloud Point, °C	-10	-8	-8
Pour Point, °C	-3	-6	-6

Table 6. Thickening Properties

Sample	Base Oil	Additive doped base oils				
		1%	2%	3%	4%	5%
P-1	BO1	16.71	11.49	9.68	8.66	8.52
	BO2	5.59	4.85	4.5	4.17	4.03
	BO3	1.79	1.67	1.49	1.35	1.3
P-2	BO1	7.8	5.64	4.99	4.43	4.39
	BO2	1.05	1.42	1.82	2.04	2.2
	BO3	0.28	0.38	0.45	0.5	0.55

Table 7. Viscosity Index Values

Sample	Base Oil	Additive doped base oils				
		1%	2%	3%	4%	5%
P-1	BO1	122.7	135.9	148.8	159.3	161.8
	BO2	118.8	130.7	137.5	146.9	154.1
	BO3	91.71	102.5	109.9	118.4	124.3
P-2	BO1	123	142.1	154.9	162.7	167.7
	BO2	118.9	131.9	141.3	150.1	157
	BO3	92.08	103	111.2	120.2	126

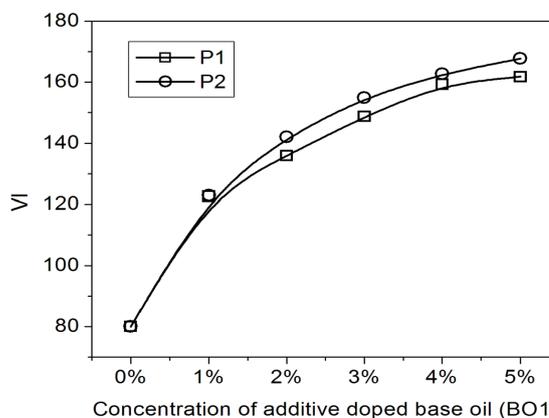


Figure 1. Comparison of VI values of the two additives in BO1 base oil

PPD properties of both the polymers (Table 7) as evaluated in different base stocks showed a good depression in the base stocks studied. It was found that the efficiency increases by increasing concentration of the additives and P-2 polymer showed better depression in pour point at higher concentrations than that of the P-1 (Figure 4 & 5). Therefore although both the polymers may be used as potential PPD for the base stocks but the polymer prepared by microwave method is better compared to thermally made polymer.

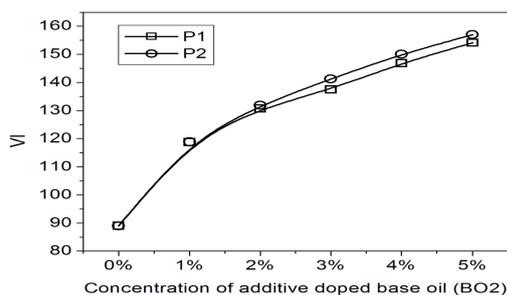


Figure 2. Comparison of the VI values of the two polymers in BO2 oil

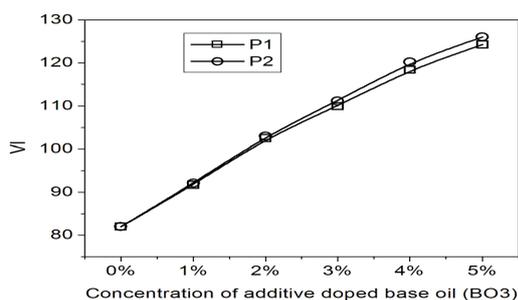


Figure 3. Comparison of the VI values of the two polymers in BO3 base oil

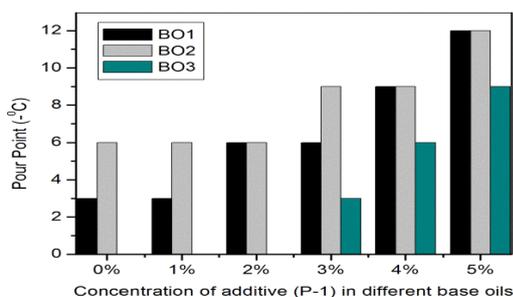


Figure 4. Pour Point of P-1 additive doped different base oils

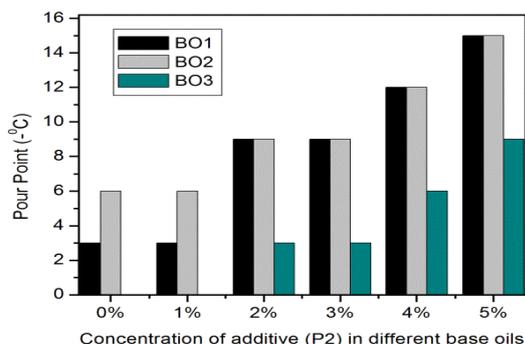


Figure 5. Pour Point of P-2 additive doped different base oils

5. Conclusions

Comparison on the basis of PPD properties, thermal stability and on the basis of determined VI of the additive blended base oil indicated that the performance of the microwave method is better than the thermal method for the additive preparation. However, both of them are found more or less equally effective as thickeners for the base oils studied. In general, it is observed that, the values of viscosity index and pour point increases with increasing concentration of the additives whereas the thickening power decreases. The studies also indicate that VI values of the additive doped base oils depend on the composition of the base oil. Comparison indicates that MW assisted synthetic method is more economical and may be considered as a greener approach for synthesis of lube oil additive.

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