

CHAPTER-IV

Multifunctional additives performance of acrylate-styrene copolymers

1.4.1. Introduction

The basic building block of a lubricant is the lubricating oil, also known as base oil or lube oil. Performance of lubricant depends on the rheological properties such as viscosity and variation of viscosity with temperature, fluidity of the oil at low temperature etc. To exhibit effective performance at both low and high temperatures, an engine lubricant should be effective fluid at low temperature and should have least variations of its viscosity with temperature. To develop the quality, lube oil normally mixed with different types of additives. Additives provide new and desirable properties to the lube oil which was not initially present in the oil or enhance the performance already existing in the lube oil.¹ The quantity and quality of the additives govern the performance of the lubricant. There are varied types of additives; such as viscosity index improvers (VII), pour point depressants (PPD), antioxidants, detergents and dispersants, corrosion inhibitors etc. Of them PPD, VII and AW are the most extensively used ingredients in modern lubricants.

Pour point depressants (PPDs)² are the chemical additives used to keep fluidity of lube oils at low temperature. They are added to continue oil flow ability below a definite temperature which is termed as pour point (PP) and well-defined as the temperature at which the flow ability of oil is just totally ceased due to wax crystal lattice formation. They are mainly polymeric hydrocarbon chains. When the temperature becomes low the oil cools down, then during the growth of wax crystal network, the hydrocarbon chains become inserted in the wax crystal lattice and thus inhibit wax crystal formation or modify the wax crystal network. Therefore, they are also known as wax crystal modifiers. Amongst the various kinds of polymers,³ acrylic and methacrylic ester polymers are very competent as crude oil wax crystal modifiers and wax deposition inhibitors.^{4,5}

Viscosity is also a very significant property of a lubricant.^{6, 7} The resistance to flow is expressed as viscosity and high viscous oil is less fluid than that of low viscosity. When the temperature is high, the oil tend to thin out and flow more easily and vice-versa. The change in viscosity with the temperature change is expressed by a parameter known as viscosity index (VI). Higher VI value of the oil reveals that viscosity of the oil differs very least with the variation of temperature. Viscosity index improvers (VII)⁸ or viscosity modifiers (VM) are added to the lubricating oil to develop the VI of the oil.

1.4.2. Experiments

1.4.2.1. Materials

Acrylic acid (stabilised with 0.02% Hydroquinone monomethyl ether) and isodecyl alcohol were obtained from Sisco Research Laboratories Pvt. Ltd. Toluene, Hydroquinone and H₂SO₄ were purchased from Merck Specialities Pvt. Ltd. Isooctyl alcohol and hexane was purchased from S D Fine Chem. Ltd. Methanol was obtained from Thomas Baker (Chemicals) Pvt. Ltd. and styrene from Across Organics. Benzoyl peroxide (BZP) purchased from LOBA chemicals was recrystallised from CHCl₃-MeOH before use. Rest of the chemicals was used as they were obtained without further purification. Two different base oils (BO1 and BO2, **Table 1.20**) were collected from IOCL, Dhakuria, Kolkata.

1.4.2.2. Esterification – preparation of monomers

Isodecyl acrylate (IDA) and isooctyl acrylate (IOA) ester were prepared by same esterification method⁹ as mentioned in the previous chapter (Chapter-II of Part I Page No. 27).

1.4.2.3. Purification of prepared esters (monomers)

The prepared esters were purified according to the procedure as mentioned in previous chapter (Chapter-II of Part I Page No. 27).

1.4.2.4. Preparation of copolymer and homopolymer

Homopolymer of IDA and IOA (HIDA and HIOA, respectively) were prepared and in the preparation of IDA + styrene and IOA + styrene copolymers, different mole fractions of styrene were used (**Table 1.21**). The homo and co polymerization was carried out by following the method as reported in the previous chapter (Chapter-II of Part I, Page No. 28).

1.4.3. Measurements

1.4.3.1. Spectroscopic measurements

To record IR spectra Shimadzu FT-IR 8300 spectrometer were used 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.

1.4.3.2. Viscometric measurements

Viscometric properties of the prepared polymers were determined at 313 K in toluene solution, using an Ubbelohde OB viscometer. Experimental determination was carried out by counting time of flow of at least eight different concentrations of the sample solution. The time of flow of the solutions was manually determined by using a chronometer. In the single point measurement, the lowest value of solution concentration was chosen for calculation.

Solution of the polymers in base oils were made by dilution method from their respective 10% stock solutions at about 60-70 $^{\circ}\text{C}$ with continuous stirring until clear solutions were obtained. Kinematic viscosities of the prepared polymer solutions were measured using Cannon-Fenske viscometer in a 100 \pm 0.1 $^{\circ}\text{C}$ bath as per ASTM D-445 procedure.

1.4.3.3. Thermogravimetric analysis (TGA)

The thermograms in air were obtained on a mettler TA-3000 system, at a heating rate of 10 K min^{-1} at room temperature under atmospheric pressure taking 0.2 g of each polymer sample

in a platinum crucible.

1.4.3.4. Evaluation of PPD properties of the additives in lube oil

The prepared additives were evaluated as pour point depressants (PPD) using two different base oils through the pour point test according to the ASTM D 97-09 on a Cloud and Pour Point Tester model WIL-471(India).

1.4.3.5. Evaluation of the prepared additives as Viscosity Index Improver (VII) in lube oil

The Viscosity index values of the polymeric oil solutions have been determined according to the ASTM D2270 method and by using the following equations.¹⁰

$$VI = 3.63(60-10^n)$$

$$\text{Where, } n = (\ln v_1 - \ln k) / \ln v_2$$

For which, v_1 and v_2 are the kinematic viscosities of the solution at lower and higher temperature respectively. The kinematic viscosity of the oil containing the different concentrations of the tested polymers was determined at 313 K and 373 K and k is a constant which is equal to 2.714 for the temperature range performed¹¹. Different concentrations ranging between 1.0 and 6.0 wt % were used to study the effect of copolymer concentration on the VI.

1.4.3.6. Evaluation of Anti-wear properties of the prepared additives in lube oil

Anti-wear properties of polymer-oil blends as well as of the mineral base stocks were studied in sliding contact by means of a Four-Ball Wear Test Machine as per ASTM D-4172.¹² The tests were carried out employing 20kg (196N) and 40kg (392N) load condition.

1.4.3.7. Determination of Average Molecular Weight

The average molecular weight of the prepared polymers was determined gel permeation chromatography (GPC). In GPC technique the number average (M_n) and weight average (M_w) molecular weights were determined using a Waters GPC system (polystyrene calibration) having molecular weight range $10^2-5 \times 10^5$ g. mol⁻¹ equipped with 2414 refractive index detector, Waters

515 HPLC pump, 717 plus auto sampler at 40⁰ C. THF has been used as an eluent at a flow rate of 1.0 ml/min at 40⁰ C. The instrument has been calibrated with polystyrene before the experiment. The poly dispersity index, indicating the nature of the distribution of the molecular weights in the polymers,¹³ was also calculated.

1.4.4. Results and discussion

1.4.4.1. Spectroscopic analysis

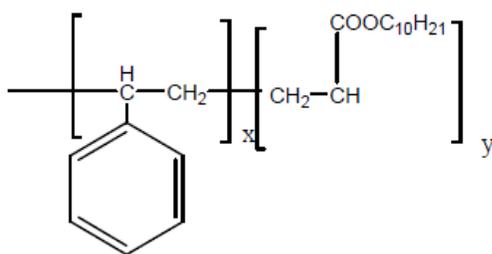
FT-IR spectrum of the homo polymer of iso decyl acrylate (HIDA) (**Figure 1.42**) showed absorption at 1732 cm⁻¹ due to ester carbonyl stretching vibration. Peak at 1260 and at 1175 cm⁻¹ can be explained owing to the C-O (ester bond) stretching vibration and the absorption bands at 975,750 and 711 cm⁻¹ were due to the bending of C-H bond. The broad peak ranging from 2900-3100 cm⁻¹ was due to the presence of stretching vibration (C-H bond). In its ¹H-NMR (**Figure 1.43**) spectra, homo polymer of IDA showed a multiplet centered at 4.016 ppm due to the proton of -OCH₂- group; a broad singlet at 0.85 ppm was due to methyl groups of decyl chain. The proton decoupled ¹³C-NMR (**Figure 1.44**) of the above sample was in complete agreement with the homopolymer which shows the presence of ester carbonyl group at 174.4 ppm and absence of any sp² carbon in the range 130-150 ppm.

FT-IR spectrum of the homo polymer of Isooctyl acrylate (HIOA) (**Figure 1.45**) exhibited absorption at 1731 cm⁻¹ due to ester carbonyl stretching vibration. Peak at 1260 and at 1164cm⁻¹ can be explained owing to the C-O (ester bond) stretching vibration and the absorption bands at 961,775 and 720 cm⁻¹ were due to the bending of C-H bond. The broad peak ranging from 2929-2950 cm⁻¹ was due to the presence of stretching vibration (C-H). ¹H and ¹³C-NMR was also in complete agreement with the homopolymer. In its ¹H-NMR spectra (**Figure 1.46**), homo polymer of IOA showed a broad singlet centered at 4.01 ppm due to the proton of -OCH₂-

group; a broad singlet at 0.89 ppm was due to methyl groups of isooctyl chain. The proton decoupled ^{13}C NMR (**Figure 1.47**) of the above sample shows no peak between 130-150 ppm which indicates the absence of any sp^2 carbon. The presence of ester carbonyl group was indicated by the peak at 170.4 ppm.

Copolymer of IDA with styrene showed FT-IR absorption (**Figure 1.48**) at 1734.72 cm^{-1} of the ester carbonyl group along with other peaks. In ^1H -NMR (**Figure 1.49**) spectra presence of peaks in the range of 7.21 to 7.55 ppm indicates the presence of a phenyl group of styrene part. The absence of singlets between 5 and 6 ppm directed the absence of vinylic protons in the copolymer. The proton decoupled ^{13}C NMR spectrum (**Figure 1.50**) showed the presence of aromatic sp^2 carbons at 126.39 and 128.36 ppm and the ester carbonyl carbon at 174.78 ppm.

The proton decoupled ^{13}C NMR spectrum of the above sample of copolymer was in complete agreement with the proposed structure of the copolymers (structure-I)



The amount of incorporation of styrene in the polymer chain (**Table 1.21**) was evaluated through a comparison of area of $-\text{OCH}_2$ group in the area of signal due to phenyl protons based on earlier reports ¹⁴ as well as on the basis of our earlier paper, ¹⁵ which was further verified through an analysis of FT-IR spectral data following a method as also discussed in our earlier paper. ¹⁵

Copolymer of IOA with styrene showed FT-IR absorption (**Figure 1.51**) at 1732.10 cm^{-1} of the ester carbonyl group along with other peaks. In $^1\text{H-NMR}$ (**Figure 1.52**) spectra presence of peak at 6.857 ppm indicates the presence of a phenyl group. The $^{13}\text{C NMR}$ spectrum (**Figure 1.53**) showed the presence of aromatic sp^2 carbons in the range 125.32 to 132.31 ppm and the ester carbonyl carbon in the range of 165.96 to 170.8 ppm.

1.4.4.2. Thermogravimetric analysis

A comparison between the TGA data (**Figure 1.54**) for homo and copolymers shows that in case of IDA polymers, the copolymers are better in thermal stability than the respective homopolymers and with increasing concentration of styrene in the feed, the stability increases. Same trend is also observed for the homo and copolymers of IOA. In addition, comparison among the TGA values indicates that the IDA polymers (homo and co) are thermally more stable than the IOA polymers.

1.4.4.3. GPC molecular weight

As expected, the molecular weight (M_w and M_n) of the homo and copolymers of IDA (M_w and M_n of HIDA is 21×10^4 and 10.5×10^4 and those for IDA + Styrene copolymer 23×10^4 and 12.4×10^4 respectively) is always greater than those of the IOA polymers (M_w and M_n of HIOA is 13.5×10^4 and 6.5×10^4 and those for IOA + Styrene copolymer 8.5×10^4 and 17.2×10^4 respectively). Lower poly dispersity index (PDI) of the IDA polymers (PDI of HIDA is 1.2 and that for IDA + Styrene copolymer is 1.8) in comparison to IOA polymers (PDI of HIOA is 3.37 and that for IOA + Styrene copolymer is 2.11) indicated lesser extent of branching in the IDA polymers. Thus a better surface related additive performance is anticipated.

1.4.4.4. Efficiency of the prepared polymers as pour point depressant (PPD)

The PPD properties of the prepared polymers were tested by using 1- 6 % (w/w) polymer

doped base oils and the experimental data are grouped in **Table 1.22**. The data indicated that the prepared additives may be considered as efficient pour point depressants. The results for most of the polymers indicate that their efficiency as PPD go on increasing from 1 % to 5 %. However, beyond the concentration of 5% the change is not significant. It is also observed that the styrene copolymers are more efficient than the respective acrylate homo polymers and the increase in styrene content enhances the performance. Also, homopolymer and copolymer of isodecyl acrylate acts as a better PPD than homo and copolymers of isooctyl acrylate.

1.4.4.5. Efficiency of the prepared compounds as viscosity index improvers

VI improver properties of the prepared homo and copolymers in terms of their viscosity index values are presented in **Table 1.23** in two different base oils BO1 and BO2. The data obtained clearly shows that the homopolymers (HIDA and HIOA) is of having lower VI values than their respective styrene copolymers and with the increase in styrene content in the stock, the VI is found to increase. Again, homopolymer of IDA and its copolymer with styrene are of having higher VI values than the respective IOA homo and copolymers, and with the increase in styrene content in the stock, the VI value is found to increase irrespective of the nature of the base oils. Again, with increasing concentration of the polymer solutions, VI value is found to increase in all the cases. This may be because of the fact that, at a higher temperature, while the lube oil viscosity gets decreased, the polymer molecules change from tight coil to expanded ones as a result of increase in the interaction between the polymer chain and the base oil.¹⁶ This increase in volume results in an increase in the viscosity of the mixture and counter balances the normal reduction in viscosity of the oil with increasing temperature. The increase in concentration of the polymer also leads to an increase in total volume of polymer coils in the oil

solutions as was already reported.¹⁷ As a consequence, a high concentration of polymer will impart a high viscosity index rather than its low concentration.¹⁸

1.4.4.6. Efficiency as anti-wear additives

Employing a Four Ball Wear Test Machine as per ASTM test conditions, the anti-wear contributions of the homo and copolymers due to the additive concentration / viscosity of the oil and due to the additive chemistry was determined. Experiments were conducted first with pure base oils followed by polymer-oil blends. The wear scar diameter (WSD) measured in all these cases are depicted in **Table 1.24** and **Figure 1.55**.

1.4.4.7. Effect of viscosity on AW performance

WSD (lower the WSD data, higher the AW benefit), is found to decrease with the viscosity of the pure base oil. The WSD data in the case of polymer - oil blends is also found to decrease with the additive concentration in the base oil i.e. with the viscosity of the polymer doped base oil irrespective of the nature of the base oils. This observation is in agreement with the earlier reported data.¹⁹

1.4.4.8. Effect of Additive Concentration on AW performance

Decrease in WSD (mm) with increasing concentration of the additives was found in both the base stocks irrespective of the types of additives (homopolymer or co-polymer). However, beyond 5% additive concentration no significant AW benefit was observed.

1.4.4.9. Effect of additive chemistry on AW performance

Acrylate-styrene copolymers always showed better AW performance in both the base oils followed by HIDA and HIOA polymers. This is due to the presence of aromatic ring of styrene in the copolymer structure, which increases the surface activity^{20,21} much more than the homopolymer samples in which the surface active phenomena is offered only by the ester

functionality present in them. It is believed that one of the oxygen atoms present in the ester group can form a donor–acceptor bond with the vacant orbital of ferrus atom using its lone pair of oxygen, thereby increasing tribological activity.^{21, 22}

1.4.5. Conclusions

- 1) IDA polymers are thermally more stable than the IOA polymers. Increased styrene content enhances the thermal stability.
- 2) Acrylate + styrene copolymers showed excellent multifunctional (PPD, VM, AW) additive performance irrespective of the nature of base oils. In general the additive performance properties of the additives, doped in the base oils, increases gradually (upto a certain limit) with the increasing additive concentration in the polymer-oil blends.
- 3) Acrylate + styrene copolymers showed much better PPD, VII, AW properties than the respective homopolymers. Homopolymer of isodecyl acrylate exhibit better additive performance than isooctyl acrylate. Again, with increasing styrene content in the copolymer, additive performance increases in all the cases.
- 4) The prepared copolymers can effectively be used as multifunctional lube oil additives.

1.4.6. References

References are given in Bibliography under Chapter-IV of Part-I (Page No. 187-189).

Table 1.20. Base oil properties

| Properties | Base oils | |
|--------------------------------------|-----------|--------|
| | BO1 | BO2 |
| Density at 313 K, Kg.m ⁻³ | 839.98 | 940.03 |
| Viscosity at 313 K, in cst | 6.808 | 22.953 |
| Viscosity at 373 K, in cst | 1.792 | 3.927 |
| Cloud point, °C | -5 | -8 |
| Pour point, °C | -3 | -6 |
| Viscosity index | 80 | 85 |

Table 1.21. Specification of prepared polymer samples and extent of incorporation of styrene

| Sample | Mole fraction of styrene in the feed | Mole fraction of styrene in the copolymer | |
|--------|--------------------------------------|---|--------------|
| | | By NMR method | By IR method |
| P-1 | 0.0000 | 0.0000 | 0.0000 |
| P-2 | 0.0155 | 0.0148 | 0.0146 |
| P-3 | 0.0425 | 0.0408 | 0.0411 |
| P-4 | 0.0711 | 0.0692 | 0.0701 |
| P-5 | 0.0000 | 0.0000 | 0.0000 |
| P-6 | 0.0135 | 0.0123 | 0.0126 |
| P-7 | 0.0391 | 0.0361 | 0.0369 |
| P-8 | 0.0637 | 0.0610 | 0.0611 |

P-1 = homopolymer of isooctyl acrylate; P-2 to P-4 = Copolymer of isooctyl acrylate + different mole fraction of styrene; P-5 = homopolymer of isodecyl acrylate; P-6 to P-8 = Copolymer of isodecyl acrylate + different mole fraction of styrene.

Table 1.22. Dependence of pour point (PP) in °C on the concentration of additives in base oil BO1 and BO2

| PP in presence of | | | | | | | | | | | | | | | | |
|-------------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Conc. | BO2 | | | | | | | | BO1 | | | | | | | |
| | P1 | P2 | P3 | P4 | P5 | P6 | P7 | P8 | P1 | P2 | P3 | P4 | P5 | P6 | P7 | P8 |
| 0 % | -3 | -3 | -3 | -3 | -3 | -3 | -3 | -3 | -6 | -6 | -6 | -6 | -6 | -6 | -6 | -6 |
| 1 % | -3 | -9 | -12 | -15 | -9 | -15 | -18 | -18 | -9 | -15 | -18 | -18 | -18 | -21 | -24 | -21 |
| 2 % | -6 | -9 | -12 | -15 | -12 | -18 | -21 | -24 | -9 | -15 | -15 | -18 | -21 | -24 | -27 | -27 |
| 3 % | -6 | -6 | -12 | -15 | -12 | -18 | -21 | -24 | -12 | -12 | -12 | -15 | -21 | -21 | -24 | -24 |
| 4% | -6 | -12 | -15 | -18 | -12 | -18 | -24 | -24 | -9 | -12 | -12 | -18 | -18 | -24 | -24 | -24 |
| 5% | -6 | -12 | -15 | -18 | -15 | -18 | -27 | -27 | -12 | -15 | -15 | -18 | -18 | -24 | -27 | -27 |
| 6% | -6 | -12 | -15 | -18 | -15 | -18 | -27 | -24 | -12 | -12 | -15 | -18 | -21 | -24 | -27 | -24 |

Table 1.23. Dependence of VI on the concentration of additives in base oil BO1 and BO2

| VI in presence of the polymer blended base oils | | | | | | | | | | | | | | | | |
|---|---------------|-----|-----|-----|-----|-----|-----|-----|---------------|-----|-----|-----|-----|-----|-----|-----|
| Conc. | Base oil, BO1 | | | | | | | | Base oil, BO2 | | | | | | | |
| | P1 | P2 | P3 | P4 | P5 | P6 | P7 | P8 | P1 | P2 | P3 | P4 | P5 | P6 | P7 | P8 |
| 0% | 85 | 85 | 85 | 85 | 85 | 85 | 85 | 85 | 80 | 80 | 80 | 80 | 80 | 80 | 80 | 80 |
| 1% | 96 | 98 | 102 | 105 | 98 | 102 | 109 | 118 | 86 | 89 | 95 | 101 | 88 | 92 | 99 | 108 |
| 2% | 95 | 101 | 108 | 108 | 103 | 107 | 112 | 121 | 90 | 93 | 98 | 103 | 98 | 99 | 109 | 115 |
| 3% | 99 | 104 | 113 | 110 | 111 | 118 | 121 | 128 | 93 | 101 | 111 | 112 | 105 | 112 | 110 | 118 |
| 4% | 104 | 111 | 115 | 114 | 117 | 122 | 124 | 132 | 99 | 105 | 114 | 120 | 110 | 113 | 117 | 121 |
| 5% | 108 | 116 | 121 | 117 | 120 | 129 | 131 | 135 | 105 | 107 | 113 | 118 | 112 | 117 | 124 | 127 |
| 6% | 112 | 119 | 120 | 100 | 130 | 133 | 132 | 137 | 108 | 113 | 118 | 124 | 114 | 122 | 126 | 131 |

Table 1.24. Anti-wear data (WSD) in BO1 and BO2 at 20kg and 40 kg load

| Wear scar diameter (mm) in presence of (BO1) | | | | | | | | |
|--|--------------|-------|--------------|-------|--------------|-------|--------------|-------|
| Concentration (%) | 20 kg. load | | | | 40 kg. load | | | |
| | HIOA-Styrene | HIOA | HIDA-Styrene | HIDA | HIOA-Styrene | HIOA | HIDA-Styrene | HIDA |
| 0 | 1.116 | 1.116 | 1.116 | 1.116 | 1.123 | 1.123 | 1.123 | 1.123 |
| 1 | 0.981 | 0.992 | 0.961 | 0.978 | 0.991 | 1.072 | 0.976 | 1.018 |
| 2 | 0.920 | 0.972 | 0.917 | 0.956 | 0.956 | 0.998 | 0.965 | 0.986 |
| 3 | 0.877 | 0.893 | 0.859 | 0.929 | 0.897 | 0.975 | 0.909 | 0.938 |
| 4 | 0.815 | 0.837 | 0.802 | 0.882 | 0.865 | 0.937 | 0.882 | 0.911 |
| 5 | 0.744 | 0.812 | 0.766 | 0.856 | 0.817 | 0.887 | 0.788 | 0.874 |
| 6 | 0.754 | 0.801 | 0.761 | 0.801 | 0.784 | 0.868 | 0.775 | 0.841 |

| Wear scar diameter (mm) in presence of (BO2) | | | | | | | | |
|--|--------------|-------|--------------|-------|--------------|-------|--------------|-------|
| Concentration (%) | 20 kg. load | | | | 40 kg. load | | | |
| | HIOA-Styrene | HIOA | HIDA-Styrene | HIDA | HIOA-Styrene | HIOA | HIDA-Styrene | HIDA |
| 0 | 0.981 | 0.981 | 0.981 | 0.981 | 0.997 | 0.997 | 0.997 | 0.997 |
| 1 | 0.969 | 0.971 | 0.916 | 0.961 | 0.988 | 0.990 | 0.916 | 0.975 |
| 2 | 0.945 | 0.962 | 0.876 | 0.938 | 0.975 | 0.981 | 0.865 | 0.946 |
| 3 | 0.917 | 0.895 | 0.864 | 0.910 | 0.967 | 0.975 | 0.811 | 0.910 |
| 4 | 0.893 | 0.863 | 0.825 | 0.886 | 0.915 | 0.944 | 0.795 | 0.886 |
| 5 | 0.871 | 0.842 | 0.785 | 0.853 | 0.877 | 0.912 | 0.774 | 0.871 |
| 6 | 0.867 | 0.835 | 0.765 | 0.843 | 0.867 | 0.886 | 0.765 | 0.862 |

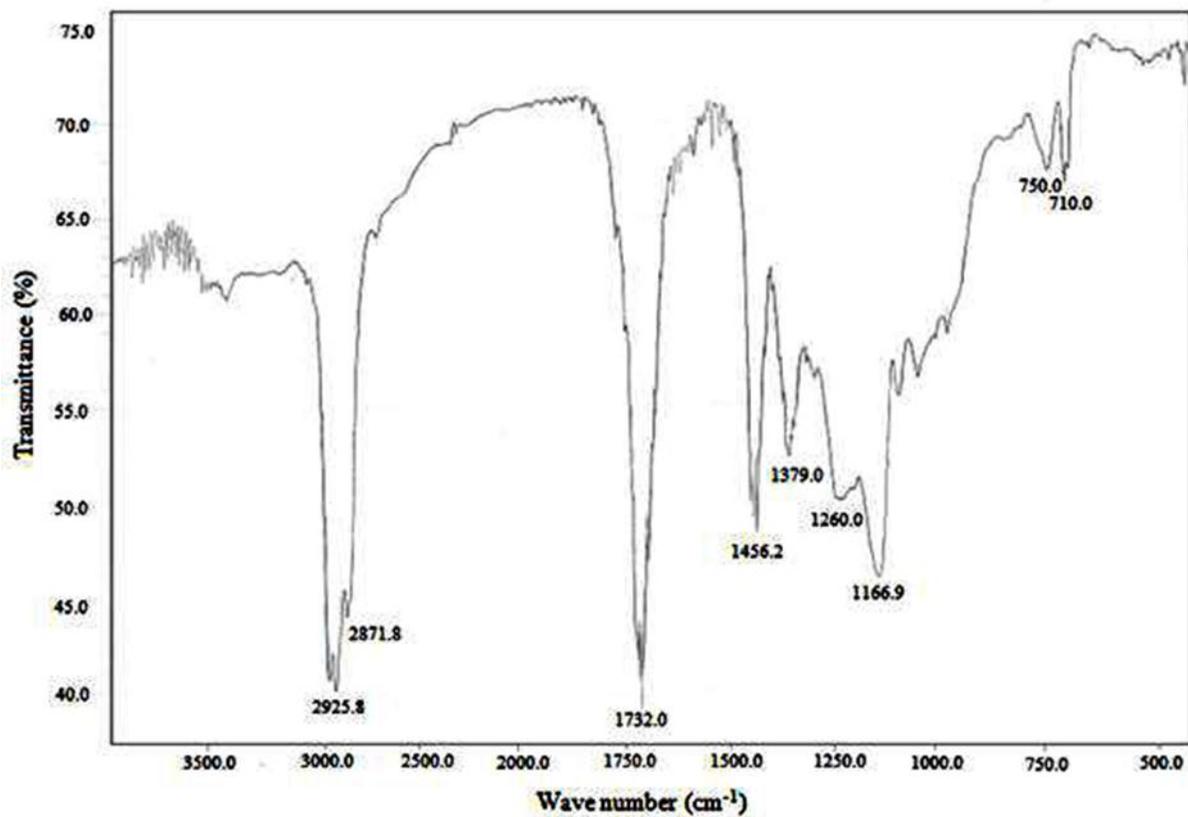


Figure 1.42. FT-IR spectra of poly (isodecyl acrylate)

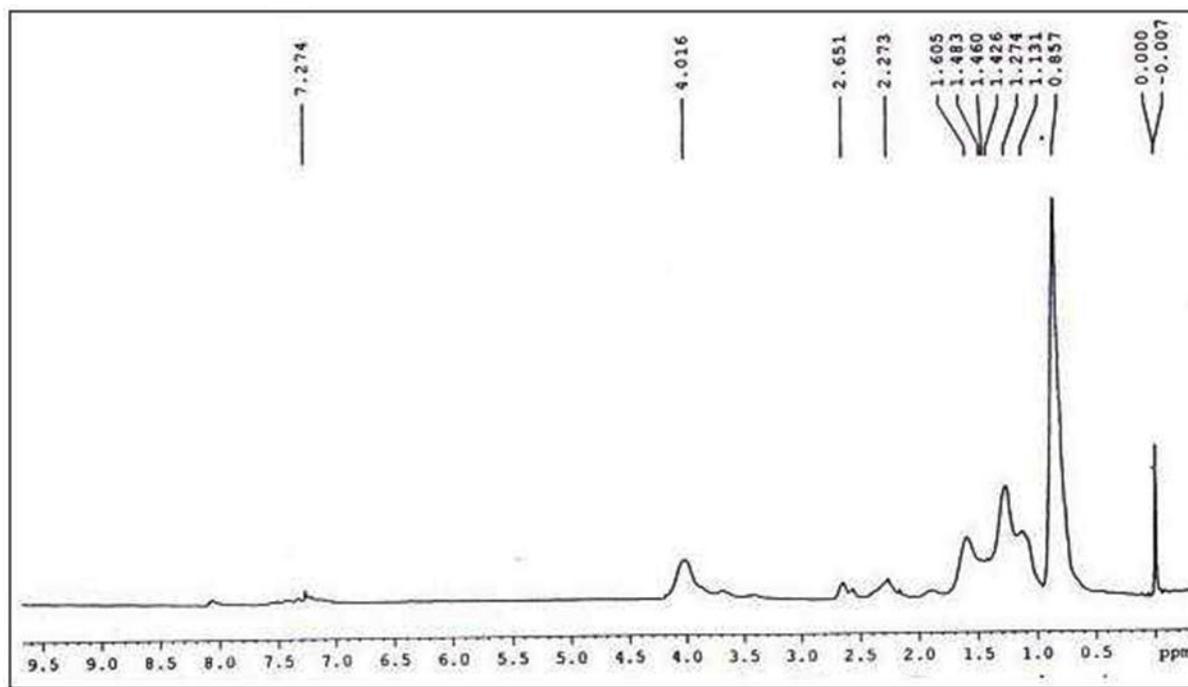


Figure 1.43. ¹H-NMR spectra of poly (isodecyl acrylate)

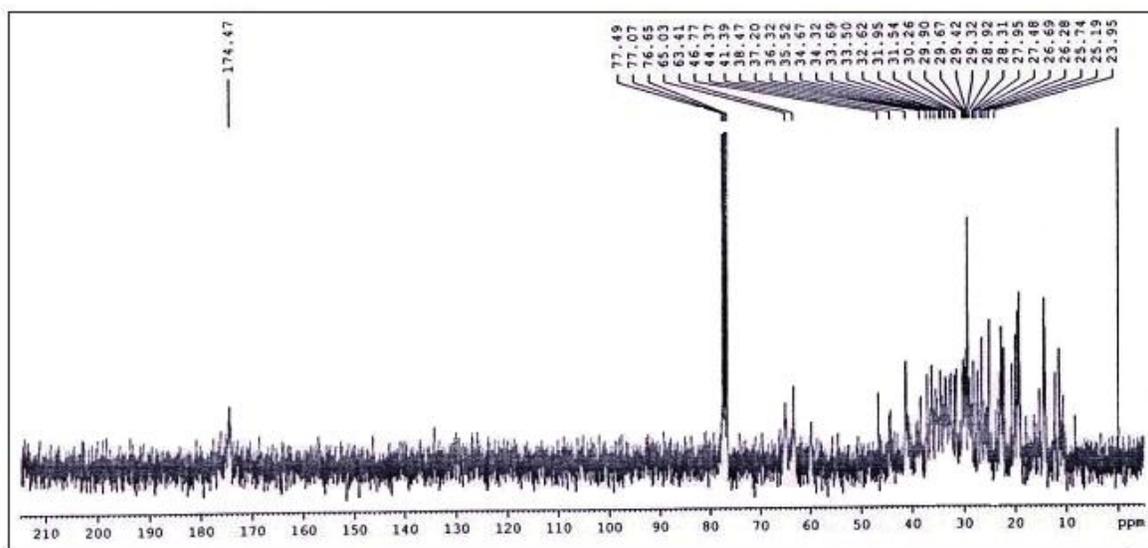


Figure 1.44. ¹³C-NMR spectra of poly (isodecyl acrylate)

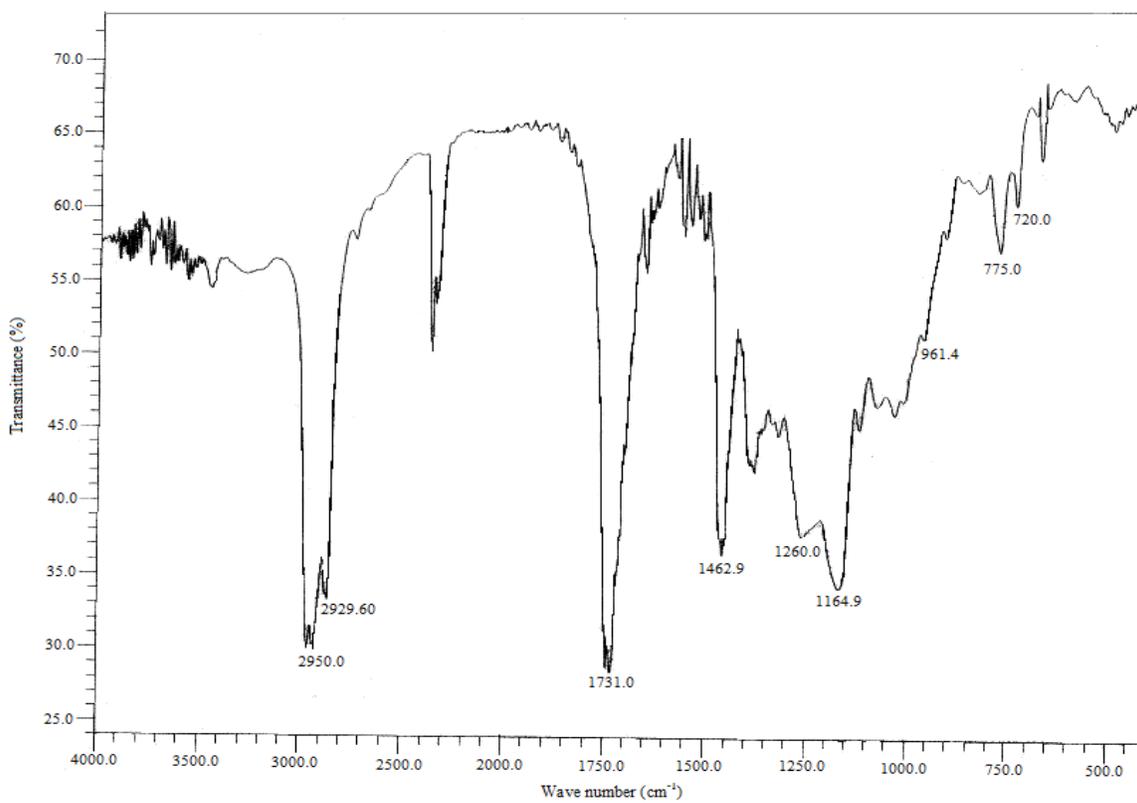


Figure 1.45. FT-IR spectra of poly (isooctyl acrylate)

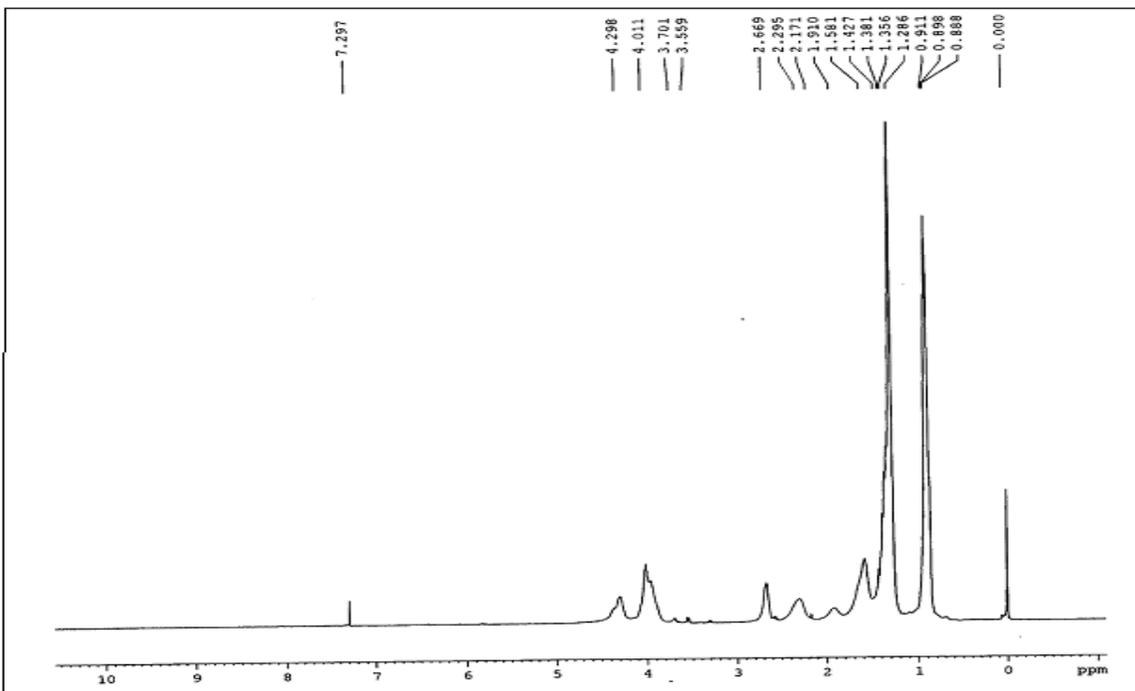


Figure 1.46. ¹H-NMR spectra of poly (isooctyl acrylate)

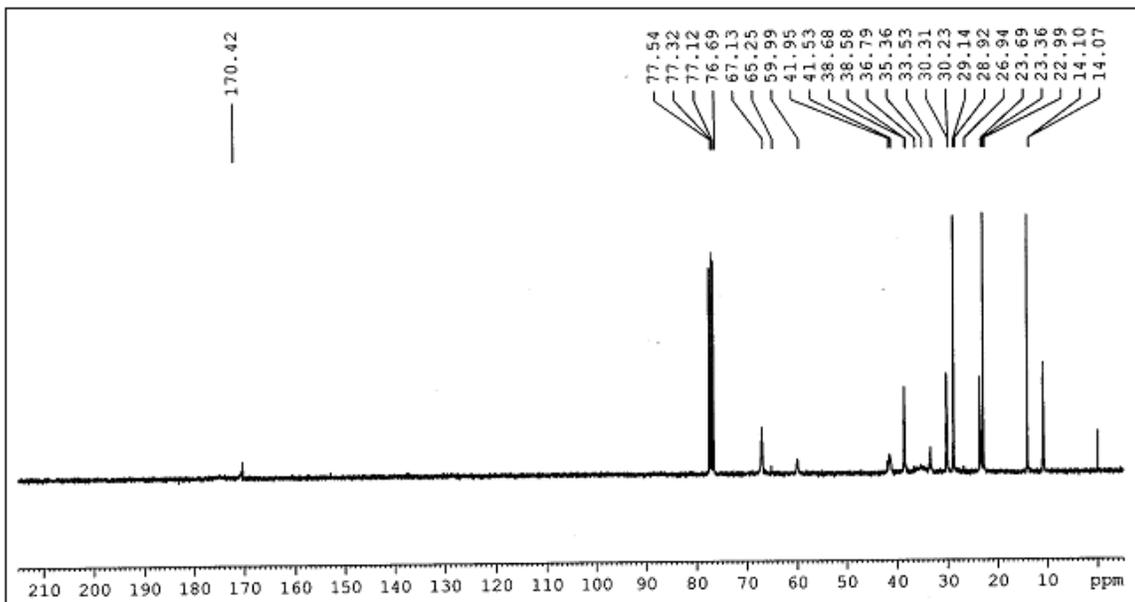


Figure 1.47. ¹³C-NMR spectra of poly (isooctyl acrylate)

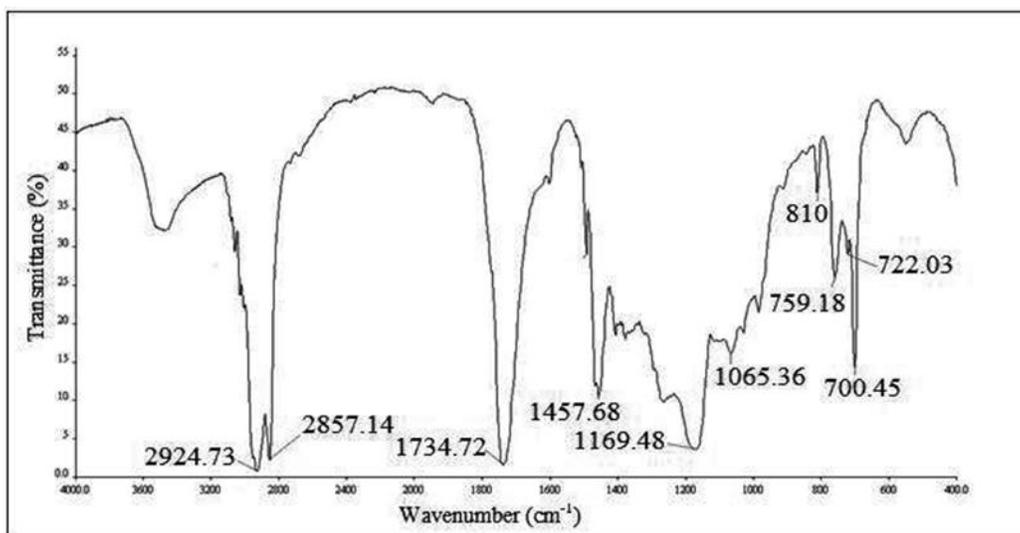


Figure 1.48. FT-IR spectra of IDA + styrene copolymer

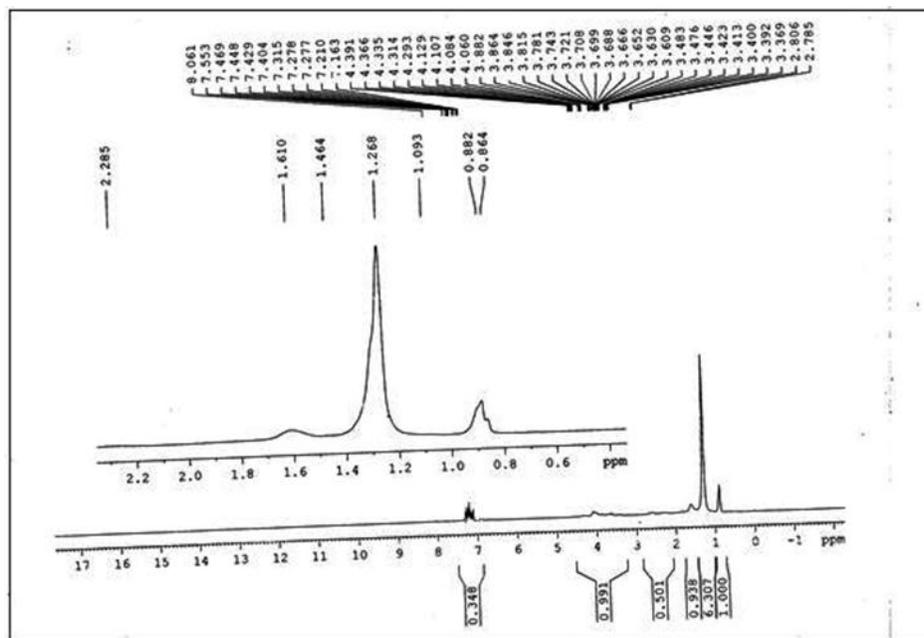


Figure 1.49. ¹H-NMR spectra of IDA + styrene copolymer

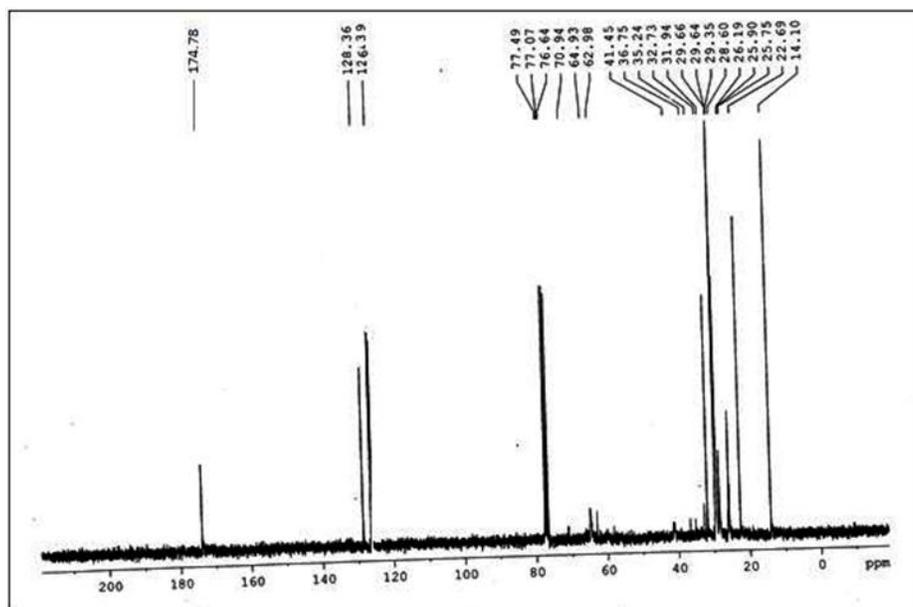


Figure 1.50. ¹³C-NMR spectra of IDA + styrene copolymer

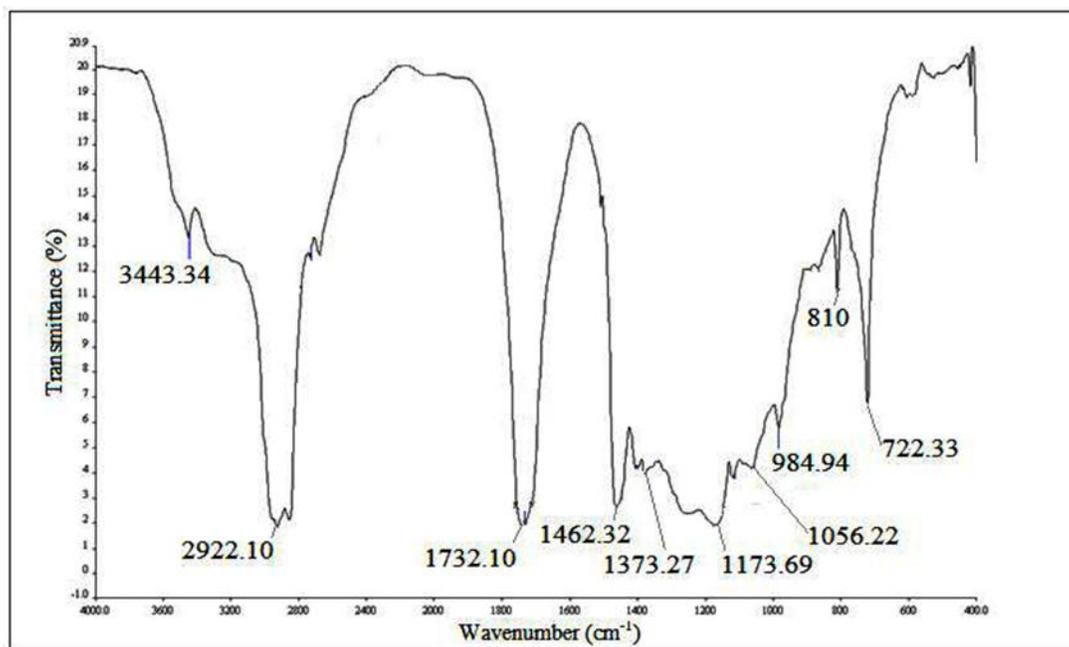


Figure 1.51. FT-IR spectra of IOA + styrene copolymer

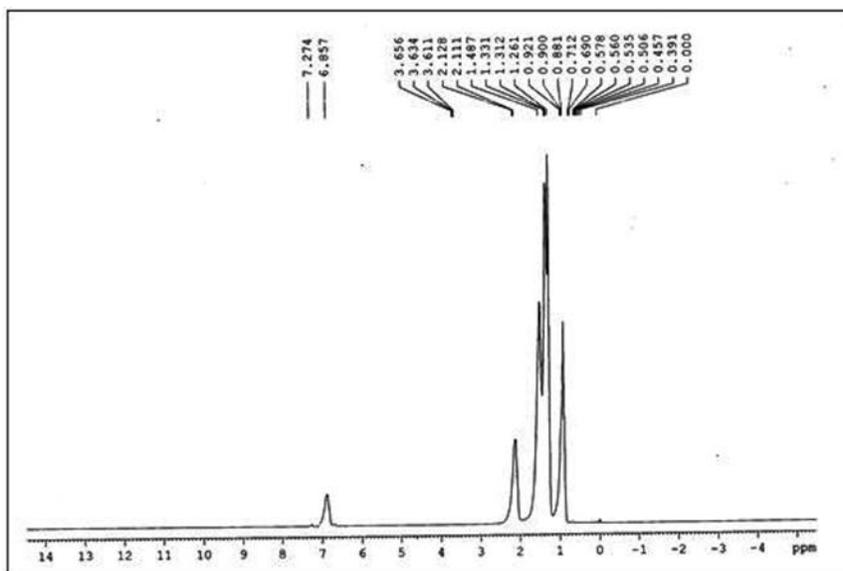


Figure 1.52. ^1H -NMR spectra of IOA + styrene copolymer

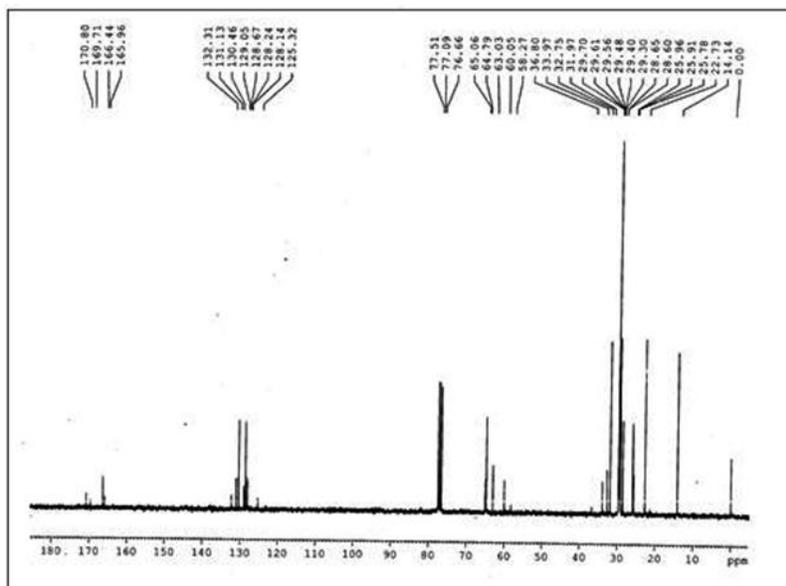


Figure 1.53. ^{13}C -NMR spectra of IOA + styrene copolymer

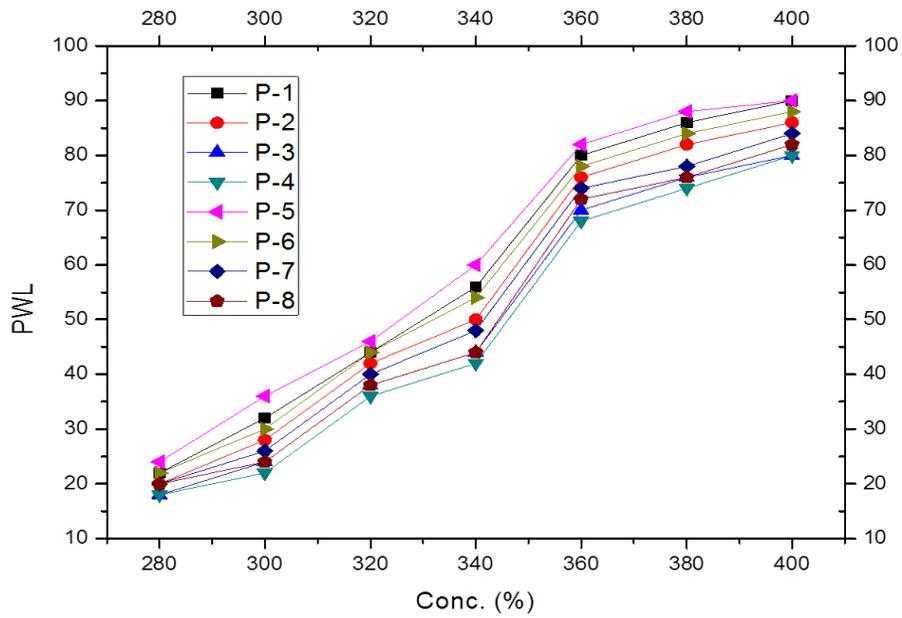


Figure 1.54. Comparison of PWL (Percent of weight loss) of prepared polymers

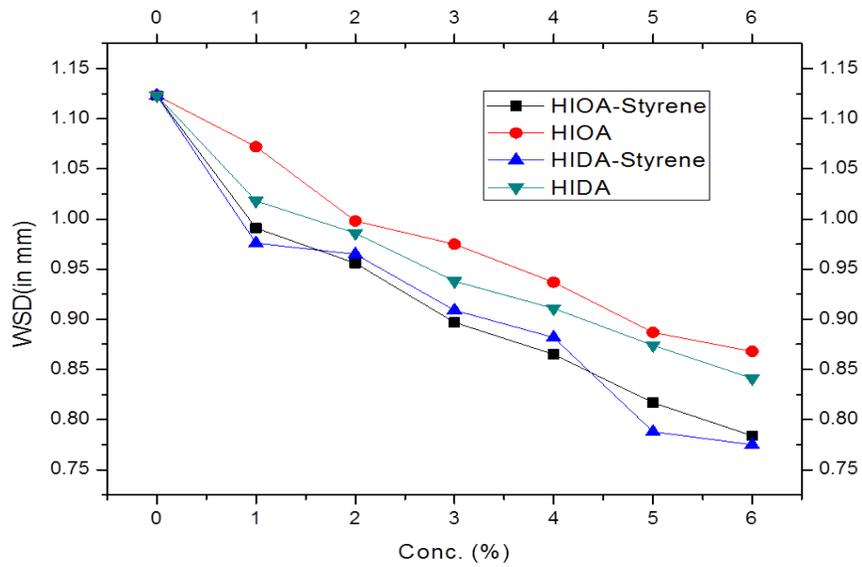


Figure 1.55. Comparison of AW property (WSD in mm) of prepared polymers in BO1 using 40 kg load.