IOSR Journal of Engineering (IOSRJEN) **www.iosrjen.org**
ISSN (e): 2250-3021, ISSN (p): 2278-8719
Vol. 04, Issue 02 (February. 2014), ||V7|| PP 20-27

Preparation, Characterization, Viscosity and Thermal Analysis of a Schiff-Based Resin derived from 2-Methoxy-1-Napthaldehyde

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Abstract: - A New Schiff based resin Poly-methylene bis (2-methyoxy-1-napthaldehyde) 1, 2-propylenediimine (PMBMNPn) was synthesized by reacting with Formaldehyde (HCHO). The synthesized resin was categorized through elemental micro-analysis and Fourier transformation. The infrared, ultraviolet and visible spectroscopic analysis TGA/DTA and Viscometric studies were also carried out. The results of Carbon-Hydrogen-Nitrogen (CHN) Analysis were in good agreement with the considered values and reinforced the formation of the prepared resin. The resin indicated more strong absorption around 16 to 35 cm^{-1} of $^{\nu}C = N$ stretching vibration than the Schiff base, confirming the structure of synthesized resin. A shift of absorption maxima towards longer wavelength within 10 to 34nm perceived in the initial 3 bands of compound PMBMNPn in comparison with BMNPn. A significant elevation in η for resin was seen as compared to Schiff base.

Keywords: *- Characterization, Resin, Synthesis, Thermo-gravimetery, Viscosity.*

I. INTRODUCTION

The Schiff based resins establish significant type of ligands which have been widely considered in coordination chemistry because of its electronic and catalytic possessions [3]. The focused resins have been utilized for pre-concentration and split-up of trace elements from ocean. Their analytical usage in combination with AAS (Atomic Absorption Spectroscopy) has well been recognized [14] and these ligands are reflected by possessing responsive functional groups of Oxygen, Nitrogen, and Sulphur in backbone of the polymer, proficient of organizing to dissimilar metal ions [12]. The structure of polymer permits favored discrimination to some metal ions and thus there is a considerable alteration in the constancy of complexes [13]. Resins of Urea-formaldehyde possess profitable position especially as glues for wooden goods and are easy for construction at cheap prices. Carbon thirteen nuclear magnetic resonance was used to study (F/U_1) mole ratio for preparing compound [9]. Thermo setting polyimide Schiff base resins find applications in electronics for making conducting or insulating thermo stable glues in the field of structural materials to prepare composite matrices [11]. Cyclohexene-formaldehyde resins can expand adhesion, rigidity and luster of covering films [16]. These can also be used as adhesives in rough coats [10]. Water proofing arrangement centered on acetone formaldehyde resin is established to elevate the efficacy of repair works to save wells from flow of water. A Salicylic acid-formaldehyde copolymer, having Orcinol (SFO), a chelating resin was found constant in acidic conditions, and was considered via Fourier transform, infrared analysis, elemental analysis, scanning electron microscopy, XRD (X-Ray Diffraction) and optical photographs [2]. Different Schiff base polymers have been produced and categorized spectroscopically and are described as arrangement of ligands [5]. The study under investigation reports the preparation, characterization, viscosity and thermal analysis of a newly formed resin, by a general method [4],[6]. These are (1) Polymethylene bis (2-methoxy-1-napthaldehyde) 1, (2) 2-diiminopropane (PMBMNPn).

II. MEASUREMENTS

The CHN (Carbon-Hydrogen-Nitrogen) Analysis was performed at Elemental Microanalysis Ltd. Devon, England, H.E.J. Research Institute of Chemistry, University of Karachi, Pakistan and NCEAC Sindh University, Jamshoro. The spectra were recorded on Nicolet Avatar 330 FT-IR (Thermo Nicolet Electron Corporation, USA) with ATR (Attenuated Total Reflectance) accessory (Smart Partner) between 4000-600 cm^{-1}. The Ultraviolet/Vis spectrums of the prepared compounds were recorded in DMF on λ 35 double beam Spectrophotometer. Quartz glass cells of one centimeter path length were used. The graphs were documented within 190nm to 700nm. The Thermo Analytical Graphs (TAG) were attained by using Pyris Diamond TGA (Thermal Gravimetric Analysis)/DTA (Differential Thermal Analysis) i.e. TA (Thermal Analyzer) from 24°C upto 600°C with a nitrogen flow rate of 5mL/min, on a heating rate of 25°C/min in an inert atmosphere. The viscosity of the prepared compound was calculated by means of the standardized Ostwald's Viscometer, with Gallen Kamp thermally controlled bath containing water. The flow time of solvent Dimethyl formamide (DMF) was recorded within 283 to 323°K with an intermission of 10°K (Kelvin).

III. EXPERIMENTAL WORK

3.1 Materials and Methods

2-methoxy-1-napthaldehyde, (Fluka, Switzerland) $CH_2NH_2CHNH_2CH_3$ (E. Merck, Germany), (DMF). $C_2H_5OC_2H_5$ and CH_3COOH (Germany), Etoh, normal-hexane (E. Merck, Germany).

3.2 Preparation of (BMNPn)

One gram of 2-methoxy-1-napthaldehyde was added to 0.198 mL of $CH_2NH_2CHNH_2CH_3$ dissoluted in 15mL of ethanol and then was refluxed for 30 minutes. The resultant oily mixture was melted in $C_2H_5OC_2H_5$ and added equivalent amount of normal-hexane. The compound crystallized and was recrystallized from ethyl alcohol. The resultant precipitates were oven dried for 4 hours at 80°C. The compound melted at 100°C. Figure-1 shows the chemical scheme for preparation of Schiff base (BMNPn).

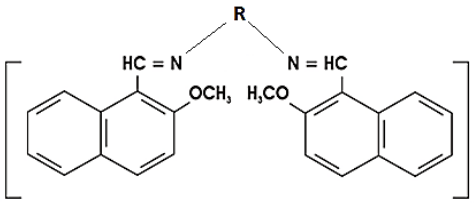


Fig.-1: Chemical Scheme for Preparation of Schiff base (BMNPn)

3.3 Poly Condensation of Schiff base with Formaldehyde to form (PMBMNPn)

One gram of BMNPn was added in 25mL of distilled H_2O, agitated the combination for 10 to 15 minutes. 15 drops of 2 molar sodium hydroxide was added thereafter. The mixture was heated upto 60°C for 5 minutes and pure solution was added 37% HCHO with it with 1:3 molar ratio. The product was boiled in bath containing oil at 120 to 130°C for 2 hours. The unsolvable resin was filtered, washed with H_2O and $C_2H_5OC_2H_5$. The precipitates were than oven dried for 7 to 8 hours at 70 to 80°C. The recovered compound was recrystallized from Etoh. The compound melted at 121°C. Figure-2 shows the chemical scheme for preparation of Resin (PMBMNPn).

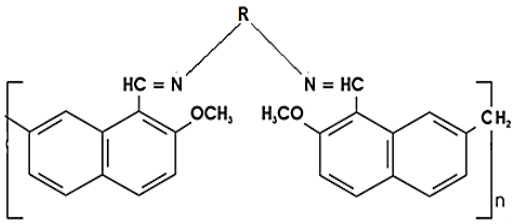


Fig.-2: Chemical Scheme for Preparation of Resin (PMBMNPn)

IV. RESULTS AND DISCUSSION

The Schiff base BMNPn, and resin PMBMNPn were prepared by a general method, and achieved in accessible yields. The results of CHN Analysis related closely to the calculated values as shown in Table-1.

Table-1: CHN Analysis of Schiff base BMNPn, and resin PMBMNPn

Compound	Chemical Formula	Melting Point	Calculated % (Found %)		
			C	H	N
BMNPn (Schiff base)	$C_{27}H_{26}N_2O_2$	95°C	78.90 (79.54)	6.38 (6.50)	6.82 (7.29)
PMBMNPn (Resin)	$(C_{28}H_{26}N_2O_2)_n$	115°C	79.40 (77.45)	6.42 (6.32)	6.61 (6.54)

The results of FTIR (Fourier Transform Infra-Red) Spectra of Schiff bases and resins are shown in Figure-3 and Figure-4. The spectra of each are sectioned into three main regions: (1) 4000 cm^{-1} to 2000 cm^{-1}, (2) 2000 cm^{-1} to 1000 cm^{-1}, and (3) 1000 to 600 cm^{-1} respectively.

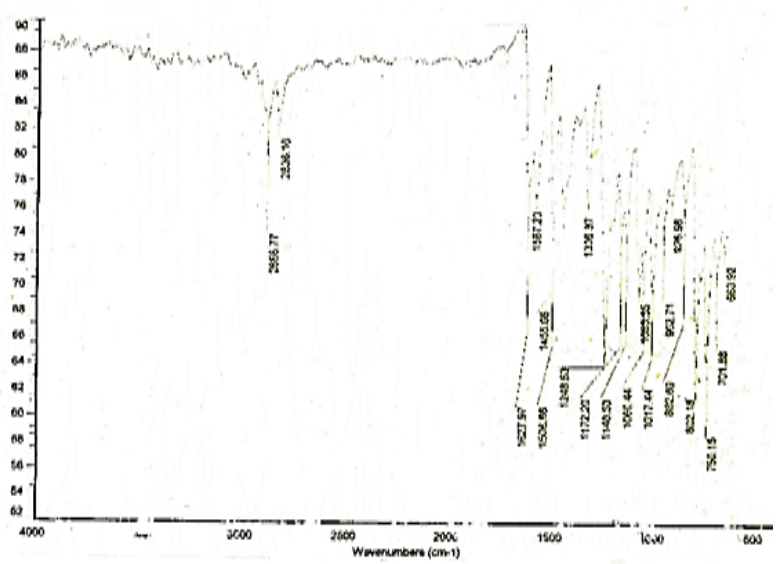


Fig.-3: FTIR Spectra of Schiff base (BMNPn)

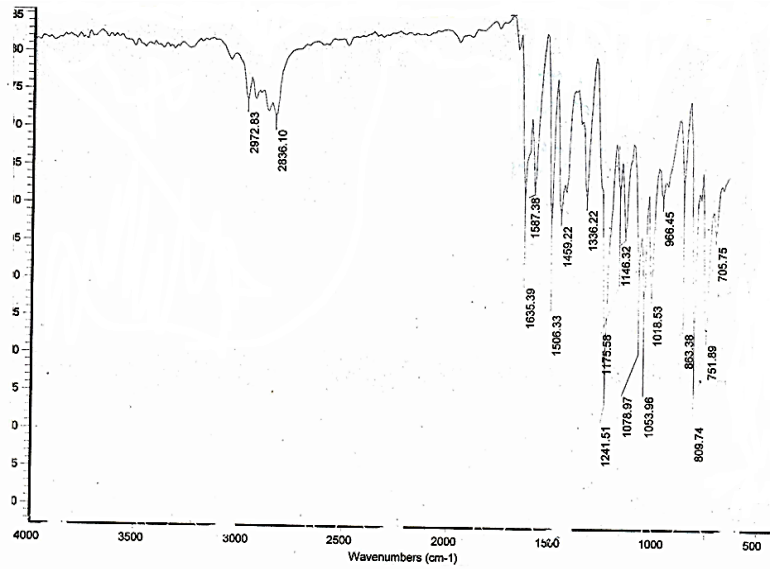


Fig.-4: FTIR Spectra of Resin (PMBMNPn)

Table-2: Results of Spectrophotometric Studies in DMF as Solvent

BMNPn	**(Schiff base)**	**λ_{max} (nm)**	270	300	348
PMBMNPn	**(Resin)**	**λ(nm), (1% ε, L·g^{-1}cm^{-1})**	280 (253.5)	330 (401.3)	382 (365.4)

The Schiff base displayed various vibrations of fluctuating intensities above 3000 cm^{-1} due to aromatic $^{v}C-H$ stretching, and the bands around 2820 cm^{-1} and 3000 cm^{-1}, due to aliphatic $^{v}C-H$ frequencies. The compound PMBMNPn at 2972 cm^{-1} and 2836 cm^{-1} show stretching vibrations due to aliphatic $^{v}C-H$ group. The resins PMBMNPn showed more stronger absorptions at 1635 cm^{-1} of $^{v}C=N$ stretching, in comparison with BMNPn that was offering comparatively medium bands thus, approving the compound's structure. The resin PMBMNPn showed a shift of 8 cm^{-1} towards higher frequency in comparison with the Schiff base, and is due to polymerization.

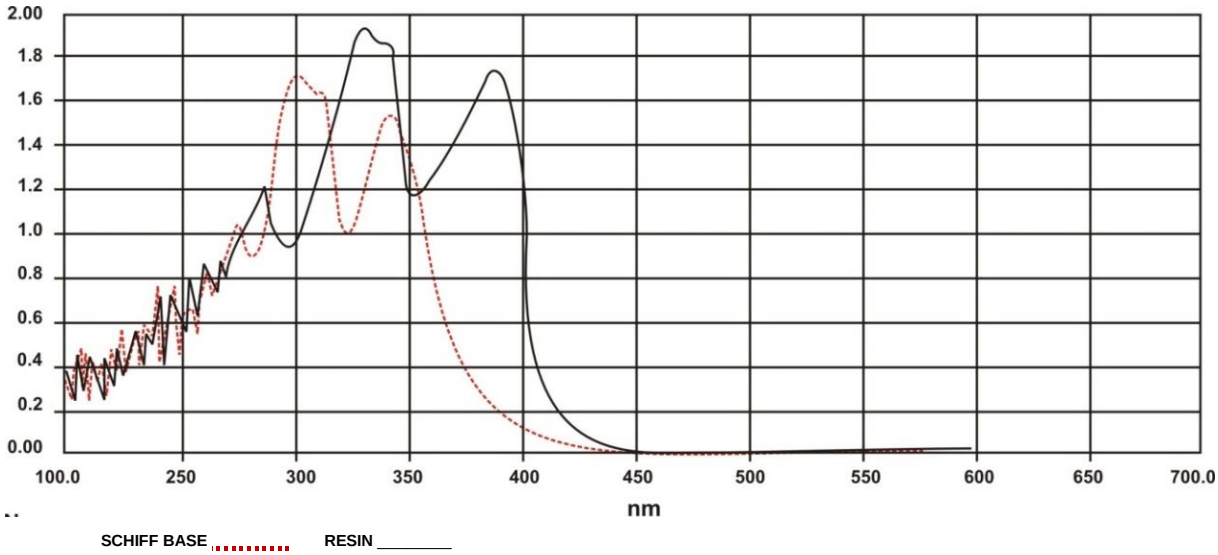


Fig.-5: The UV/Visible Absorption Spectrums of Schiff base (BMNPn) and Resin (PMBMNPn)

The UV/Visible absorption spectrums of Schiff base (BMNPn) and Resin (PMBMNPn) are shown in Figure-5. The spectrums of BMNPn showed 3 bands at 270nm, 300nm, and at 348nm with molar absorptivities 20739.0 L·mol^{-1}cm^{-1}, 34907.6 L·mol^{-1}cm^{-1}, 31211.5 L·mol^{-1}cm^{-1}, due to $\pi-\pi^*$ evolutions in aromatic napthyl ring system and due to $\pi-\pi^*$ changes concerning napthyl rings and $^{v}C=N$ π-electrons. The resin PMBMNPn, displayed absorptions at 280nm, 330nm and at 382nm with (ε 1% = 253.5 L·g^{-1}cm^{-1}, 401.3 L·g^{-1}cm^{-1}, and 365.4 L·g^{-1}cm^{-1}) due to $\pi-\pi^*$ shifts in napthyl rings and $\pi-\pi^*$ shifts relating napthyl rings and conjugated $C=N$, π-electron systems. A shift towards longer wavelengths of 10 to 34nm was perceived in the first three bands of resin PMBMNPn in comparison with Schiff base due to poly condensation as shown in Table-2 above. The thermo analytical plots were obtained by using Pyris Diamond TGA (Thermo Gravimetric Analyzer) from 24°C to 600°C with a nitrogen flow rate of 5mL/min at a heating rate of 25mL/min in an inert atmosphere [1].

Thermal analyses of the Schiff base BMNPn is shown in Figure-6, and Resin PMBMNPn is shown in Figure-7. The weight loss of about 0.8 % in the region of 50–100°C as shown in Figure-6 is due to removal of water from the sample. Within 200 and 265°C, the material faced quick weight loss, with the rate of loss 2.18% per °C at 250°C T_{max} (maximum rate of weight loss). The total or 100% weight loss is also observed at 270°C.

Two endotherms were detected in the DTA plot at 88°C and 265°C. The first agrees to the melting of the Schiff base and second with DTA peak is linked to the vaporization/volatilization whereas the resin PMBMNPn displayed first weight loss of about 2% within 50 to 100°C due to removal of H_2O from compound. Within 280 and 350°C, the compound faced fast weight loss with the degree of loss 1.97%/°C at 322°C i.e. the maximum rate of weight loss, T_{max}. The 100% weight loss observed at 500°C.

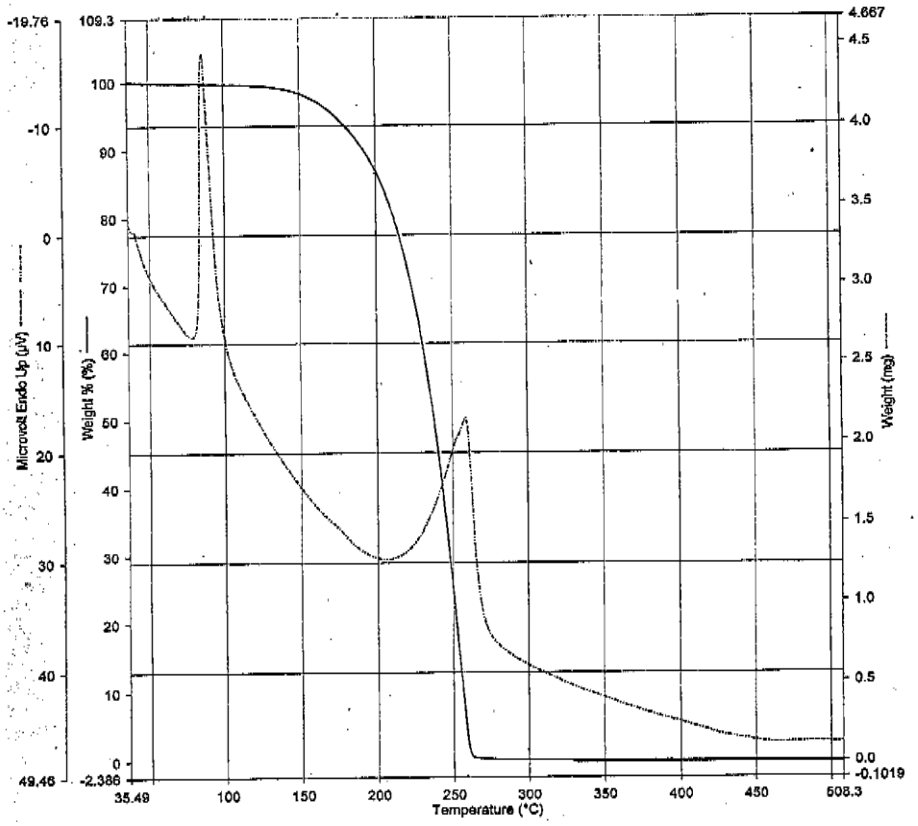


Fig.-6: Thermal Analyses of the Schiff base BMNPn

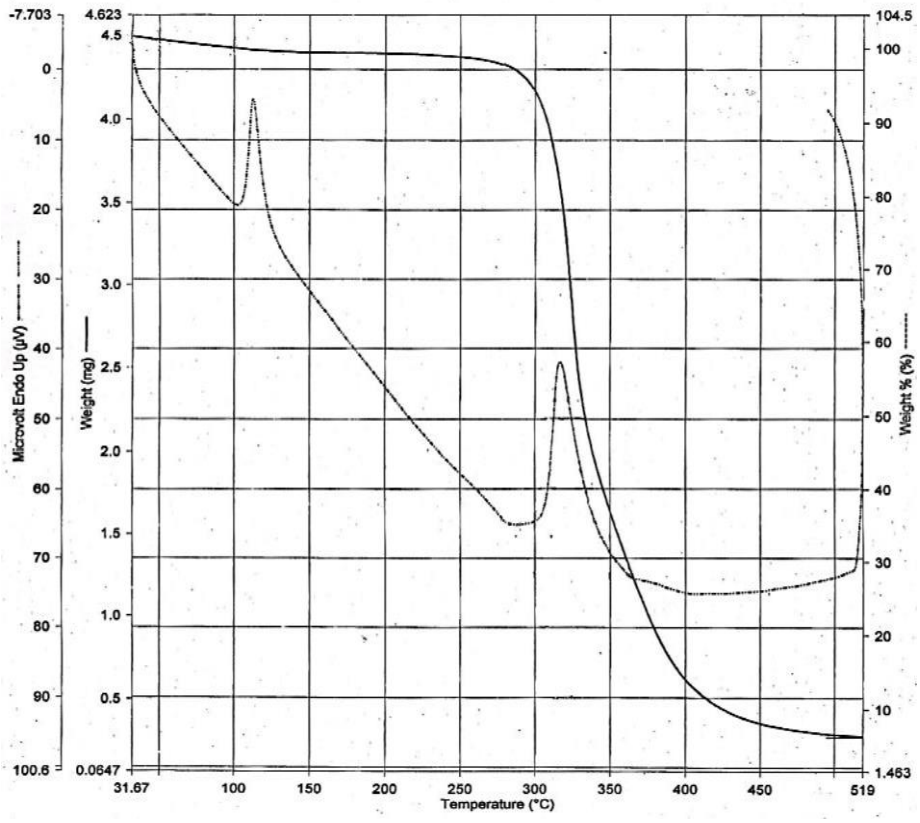
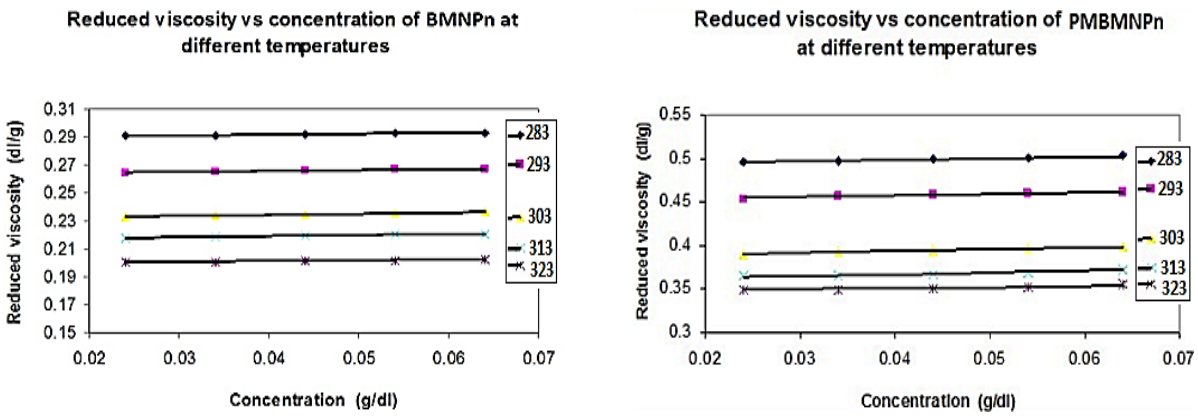


Fig.-7: Thermal Analyses of the Resin PMBMNPn



(a) Schiff base (b) Resin

Fig.-8: Reduced viscosity vs Concentration at different temperature of (a) Schiff base, (b) Resin

Two further strong endotherms were detected in the DTA curve at 115°C and 317°C. The first one agrees to the melting of the resin and the other one was related to the vaporization/volatilization/decomposition. The viscometric measurements of all compounds were calculated by using the calibrated Ostwald's Viscometer. The compounds of concentrations 0.024 to 0.064g/dL were equipped by adding Dimethyl formamide to the re-weighed Schiff base or resin sample in a volumetric flask. The viscometer was occupied with 15mL of solution with concentrations 0.024 to 0.064g/dL and a flow time at 283°K to 323°K with an interval of 10°K were measured [7]. From the flow time of the solvent and solution the parameters discussed in the beginning were calculated.

The graph-plot plotted between concentrations and $\eta_{reduced}$ as shown in Figure-8 (a) & (b) showed linear relationship obeying Huggin's equation. The reduced viscosity $\eta_{reduced}$ of Schiff base was within 0.2–0.4 dL/g and for resin 0.3–0.5 dL/g. The resin showed enhancement in reduced viscosity $\eta_{reduced}$ as compared to its Schiff base and the results indicated that values of reduced viscosity rises with the escalation in concentration. Intrinsic viscosity is directly related to the polymer sizes and as the polymeric chain increases the molecule becomes huge, resulting into a rise in intrinsic viscosity. Table-3 shows the values of intrinsic viscosity η. For Schiff base it was 0.2890–0.2015 dL/g and for resin 0.490–0.345 dL/g exhibiting that the values increases as the molecular weight increases.

Table-3: Intrinsic Viscosity values of Schiff base and Resin (dL/g)

Compounds	**Temperature °K**				
	283	**293**	**303**	**313**	**323**
BMNPn	0.2890	0.2629	0.2310	0.2160	0.2015
PMBMNPn	0.4908	0.4505	0.3860	0.3593	0.3449

The absolute viscosity η_{abs} calculated for Schiff base BMNPn was 0.199–0.430 and for resin PMBMNPn 0.286–0.464 m.N.s/m^2. The values of η_{abs} for Schiff base and Resin rises with the escalation in concentration due to rise in density of polymeric solution, and reduced with elevated temperature. The ΔG_V values of the prepared compounds rise with temperature and concentration, and were within the range for BMNPn 13.936–14.397 K.J.mol^{-1} and for resin PMBMNPn 14.383–15.279 K.J.mol^{-1}. The polymeric resin possess weaker associations and easily astounded during the process of flow, but as the concentration rises the connotations become stronger and gets least affected [8]. The increase in ΔG_V with elevation in temperature indicated conversion of shape of molecules of polymeric resin [15],[17]. The values of entropy were within –0.00447 to 0.00322g/dL and for resin –0.01965 to 0.20258g/dL. The negative values indicated order in the system.

V. CONCLUSION

A new Schiff base and a resin was synthesized, and thoroughly characterized via elemental microanalysis, FTIR, UV/VIS Thermo Analytical (TA) studies and viscometery. The CHN values of the compounds found closely correlated to the calculated values conforming the formation of compounds. The resin indicated more stronger absorption band at 1635 cm^{-1} of azomethine $^{v}C = N$ stretching vibration than the Schiff base, approving the structure of the newly prepared resin. A shift of absorption maxima towards longer wavelengths of 10 to 34nm was detected in the first three absorption bands of resin PMBMNPn as compared to Schiff base BMNPn. A significant elevation in η for resin was observed as compared to Schiff base.

VI. ACKNOWLEDGEMENT

The authors are thankful to Dr. M. A. Kazi Institute of Chemistry, University of Sindh, Jamshoro, Pakistan for providing research facilities at Analytical Chemistry Labs. The CHN analysis was conducted at Elemental Microanalysis Ltd. Carlo Elba Devon, United Kingdom, H.E.J. Research Institute of Chemistry, University of Karachi, Pakistan and NCEAC, University of Sindh, Jamshoro. The authors are also thankful to Incharge, Softronix Science Simulation Lab (FF-21, UBIT Building), Department of Computer Science, University of Karachi, Pakistan for providing support for performing data analysis, software based simulation, and document processing of this Research paper.

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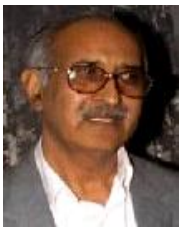

Muhammad Yar Khuhawar is Professor Emeritus, leading researcher and a versatile educator with distinguished academic carrier producing 25 Ph.D., 9 M.Phil. and 3 M.E. research scholars. He is honored with large number of National Awards, including Izaz-e-Kamal and Sitar-e-Imtiaz by President of Islamic Republic of Pakistan for achievements in scientific research. He is well known scholar having 35 years experience of teaching and research at higher education. Administrated number of fully funded research schemes and various academic tasks. National reputation for launching highly productive research workspace, in spite of meager research facilities available. He is actively committed to the field of research yielding over 283 publications in national and international journals. A large number of them are in the field of chromatography and electrophoresis for the analytical methods development for metals, non-metals and biological active compounds from wide variety of real samples; the synthesis of a number of schiff base polymers; new stationary phases for gas chromatography and sensitive liquid chromatography procedures for the determination of pesticide malathion.

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