International Journal of Chemistry and Pharmaceutical Sciences

Journal Home Page: www.pharmaresearchlibrary.com/ijcps

Research Article **Open Access**

Synthesis of Poly Vinyl Alcohol with New(S)-2-(4-Methoxyphenylamino)-2-Phenyl Acetonitrile Compound

Hizoom Mola Al-Mayiah*, Mohammed H. A. Al-Amery, Sanaa A.Sahib A.Kareem

[1]*Department of Chemistry, College of Science, Baghdad University,*
[2]*Department of Chemistry, College of Science for Women, Baghdad University,*
[3]*Department of Chemistry, College of Science for Women, Baghdad University*

A B S T R A C T

Anew synthesis of poly vinyl alcohol with adipoly chloride under refluxed for 6 hr. to give poly (vinyloxy acid chloride) then the product reacted with (S)-2- (4-methoxyphenylamino)-2-phenyl acetonitrile under refluxed for 6 hr. in presence of bonzen at 61°C to give poly fviny P(S)-2-{4-nicihoxyphenyJamino)- 2-phenyl acetonitrile. According to the obtained product the coordinate of N2- donor atoms in the compound. N-H group in the compound is very active and can enter many reactions. NMR identified the prepared polymer and FT-IR, Solubility and thermal stability were study.

Keywords: poly amino nitrile. (S)-2-(4-miethoxnphenylarnino)-2-phenyl acetonitrile.

A R T I C L E I N F O

CONTENTS

Article History: Received 09 September 2015, **Accepted** 19 October 2015, **Available Online** 27 November 2015

***Corresponding Author**
Hizoom Mola Al-Mayiah
Department of Chemistry,
College of Science, Baghdad University
Manuscript ID: IJCPS2705

PAPER-QR CODE

Citation: Hizoom Mola Al-Mayiah, *et al*. Synthesis of Poly Vinyl Alcohol with New(S)-2-(4-Methoxyphenylamino)-2-Phenyl Acetonitrile Compound. *Int. J. Chem, Pharm, Sci.*, 2015, 3(11): 2095–2098.

1. Introduction

Poly amino acids are important compounds in medicinal and pharmaceutical field. They show biological activities including anti-bacterial [1,4], anticancer [7,9]. Furthermore, this new amino polymer has been widely used as protective group of amino group in organic synthesis [11,12]. Strecker reaction provides one of the most efficient method for the synthesis of <x - aminonitrile since 1850 when used an aldehyde, amine, and HCN, addition of (CN‘) to imines provides direct reaction of ∞- aminonitrile derivatives have high fungi static activity more than commercial fungicide

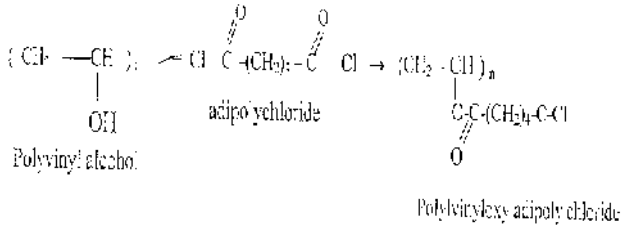
kaptan [1]. cc - aminonitriles are useful for synthesis of amino acids [2,3] among many other applications which are interest as ligand for Zinc (II) complex with potential antitumor properties [7,8]. oc - aminonitrile has often been used as chelating ligand through two nitrogen atoms in amino coordination chemistry.

2. Materials and Methods

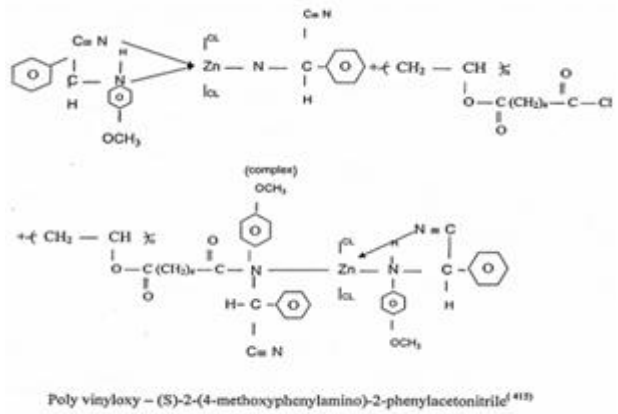
All chemicals used were analytical analar and of highest purity available from B.H.D. company. The prepared lighand (S)-2-(4-methoxyphenylamino)-2- phenyl acetonitrile was synthesized and characterized according to published work. Melting points were determined on Gallenkamp melting point apparatus (MFB-600). Structures conformation of new prepared polymer and ligand were proved by FT-IR spectroscopy using. Csl disk on shimadzu (8300) and other physical properties including solubility melting points of polymer were measured.

Preparation of Softenibg point (189-201) °C

Polylvinyloxy adipoly chloride (0.011 mole) of polyvinyl alcohol [6](PVA) and (0.011 mole) of adipoly dichloride dissolved in 7 ml of benzene and refluxed at 61°C for six hrs. Filtered and purified with T.H.F. conversion of yield = (81%). Melting point (203-205)°C. The reaction was clarified below:

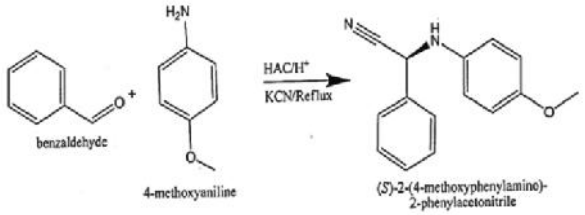


The 0.011 mole of poly vinyloxy with complex of (0.011 mole) dissolved in 7 ml benzene heated under reflux for six hrs. Filtered and purified with THF the reaction clarified below:



Preparation of (S)-2-(4-methoxyphenylamino)-2-phenyl acetonitrile ligand (L) (A13)

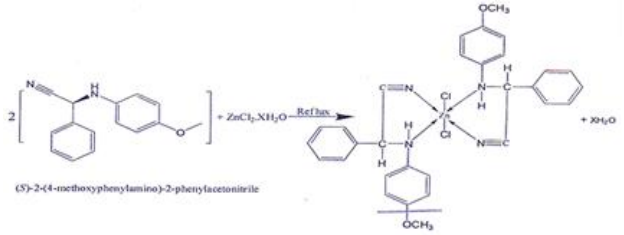
A general method [15,16] can be adopted for [L] preparation which can be described as follows: The benzaldehyde (1 mmol) was added to (20 ml) glacial acetic acid, p-toluene sulphonic acid was added in very small portion as catalyst, followed by addition of (1 mmol) p-methoxy aniline. The pH was adjusted to about 4 by addition of concentrated sulfuric acid drop wise to obtain Schiff base which stirred for 30 min. Potassium cyanide (2 mmol) was added to the mixture and kept under stirring for 4 days. The reaction mixture was poured into ice and then made slightly alkaline with ammonium hydroxide solution. The solid precipitate, which formed, was filtered, washed with water and air-dried. The presence of nitrile group in the prepared a-aminonitrile was indicated.



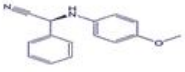
By treating few amount of the sample with 10% sodium hydroxide solution, the liberation of ammonia after hydrolysis of nitrile group, which was detected by wet red litmus paper, indicating the presence of nitrile group. Purity of the obtained compound was checked by TLC, using chloroform and ethyl acetate (1:1) as eluent. The product color was light green and the yield percentage was (85.23 %) and the melting point was (58-60 °C). The reaction was clarified below:

Preparation of metal complex:

The complexes [Znl^Cy.EhO have been prepared by the reaction of (0.476 gm) (2 mmol) of (L) with (1 mmol) of Zinc chloride (0.136 gm of ZnCf) dissolved in (20 ml) absolute ethanol and refluxed with stirring under anhydrous conditions using Na_2S0_4 (anhydrous) for 24 hours. The obtained complex was collected after evaporation of ethanol and triturated with petroleum ether (60-80 °C) then filtered and the product was left in the desiccators to be dried under P_2O_5. The general reaction was clarified below:



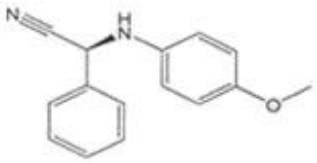
Note:- [L = A13



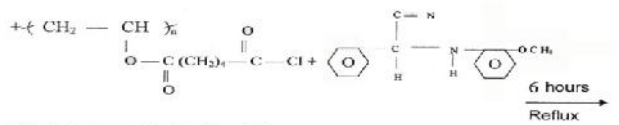
Preparation of poly (vinyloxy- -4-yl amino)phenyl acetonitirile [4,5]: (1 mmole) of benzaldehyde was added to 20 ml of glacial acetic acid, P- toluene sulphonic acid was added as catalyst in d by addition of (1 mmole) P-methoxy aniline at PH=4 by addition of conc. Sulphuric acid drop wise to obtain Schiff base which stirred for 30 min. Potassium cyanide (2 mmole) was added to the mixture and kept under stirring for 4 days. Mixture was poured into icc and then made slightly alkaline with ammonium hydroxide solution precipitate was formed and filtered, washed with water and air dried.

Nitrile group in the prepared oc- aminonitrile was indicated by treating with few amount of 10% NaOH solution liberation of ammonia after hydrolysis of nitrile group. Which was detected by wet red litmus paper indicating the presence of TLC, using CHC13 and CH_3CH_2 C- OCH_3 (l:l) as eluent. The product color was light green and percentage yield was (85.23%), melting point was (58-60)°C. The reaction was clarified below. Reaction of 0.01 l mole poly (vinyloxy adepoly chlovide) with 0.011 mole of (N -3-methoxy phenylamino).

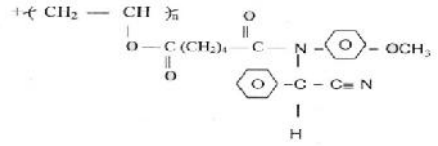
Phenyl acctonitrile



In 7 ml of benzene under refluxed with THF conversion yield (75%) softening point (189-201)°C and melting point (203 -205) °C.' The reaction was clarified below:

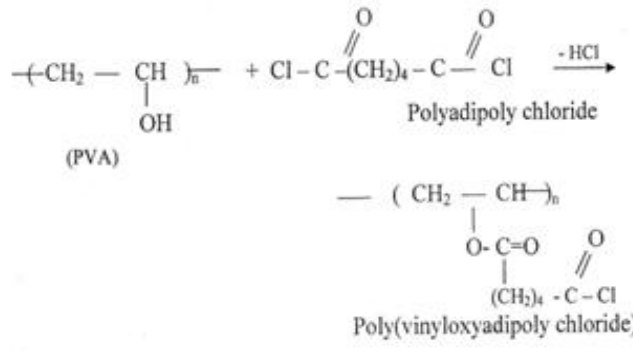


Poly (vinyloxy adipoly chlovide)



3. Results and Discussion

One of suitable procedure for preparation of poly (vinyl alcohol from (vinyl acetate) by hydrolysis in acidic medium with acetone under reflux. Physical properties in table (1-1). The FT-IR spectrum show absorption band at (32500-3600) cm^{-1} for OH- group and at 1250 cm^{-1} for O-C-O alcohol and for - CH aliphatic at 2990 cm^{-1}. The FT- IR spectra for poly (vinyloxy adipoly chloride), show absorption band at 1280 cm"1 for o-c-o ester group and at 1697 cm'1 for C=0 group and at 2916 for - CH group and at 617 cm'1 fori C- Cl), mechanism reaction in scheme (l).



All compounds reported in this work in table (1) which illustrated physical properties, along with molar conrductivity at room temp. Metal and elemental analysis of all compound are represented in table (2).(N-4- methoxy phenyl) aminophenyl acetonitrile was prepared as reported in literature [17]. It was characterized by elemental analysis and infrared spectral data. The complexes have been prepared under anhydrous conditions to avoid any hydrolysis of nitrile group to amide duo to the presence of metal ion <[18]> OC- amino are useful inter mediates for synthesis of amino acid [2,3], N- containing heterocycles such as thienopyrimidine derivatives which have antibacterial and antifungal activities higher than the cores ponding antibacterial (ampicillin and antifungal nystatin) [4-6]. Moreover , they are readily hydrolyzed to diamine , which are interest as ligands for Pt(ll) complex with potential antitumor properties oc- aminonitrile have been used as chelating ligand through two nitrogen atoms in field of amino coordination chemistry. This is new study of coordination field as ligand with metals ions. These complexes were stable in solid state and characterized by molar conductivity, metal and elemental analysis, magnetic susceptibility, FTIR and UV- visible spectroscopy , it's suggested that all complexes have- structure with coordination [6] No. that includes two atoms of coordinate chlorides. Conductivity for the complexes $10'^{3}$M in absolute ethanol in rang [4-12] indicating non- electrolytic nature of complexes [20]. Moleration used to determine the ratio of metal ion to ligand complexes at T_{max}(T_{7naz} = 571 *nm for [Col$_2$cl$_2$]and* /Ima* = 460*nm for [Cul$_2$cl$_2$\ .2 H$_2$0]* in alcoholic solution.

Table (2): Infrared absorption band $(cm)^{-1}$ of new prepared polymers

comp ound No.	compound name	ν_{C-O} ν_{C-O} acid - OH	$\nu_{C=O}$ ν_{acid} ester	ν_{C-OC} cm^{-1}	ν_{C-H} bendig cm^{-1}	$\nu_{C=C}$ aram atic	ν_{C-H} aliph atic cm^{-1}	ν_{C-H} aroatic cm^{-1}	Other s bands cm^{-1} - OH	ν_{C-Cl}
1	Poly (vinyl alcohol)	—— 1250	-	-	1459	-	2990	-	3250-3600	
2	poly adipoy chloride	1218	1650	1280			2916			617

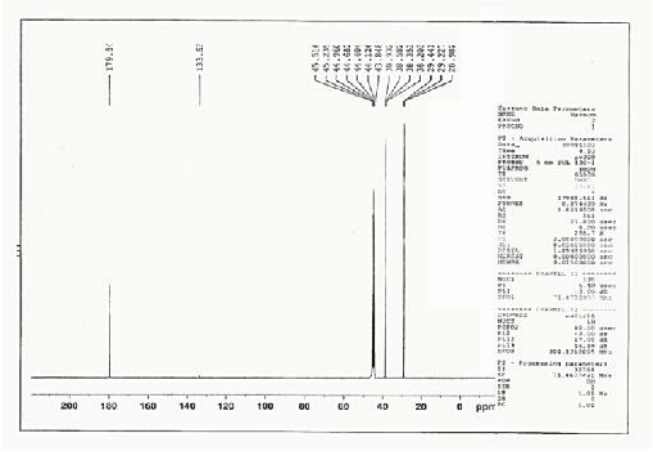
Table (3): Solubility of new polymer

No.	Benzene	DMF	DMSO	THF	Water	CCl4	Acetone	EtOH
poly (vinyl alcohol)	VS.	VS.	VS.	VS.	ps.	ps.	VS.	VS.
poly (vinyloxy-adipoly chloride)	VS.	VS.	VS.	VS.	ps.	ps.	VS.	VS.

vs. = very soluble ps. = partial soluble

Table (4): ^{1}H-NMR spectra of selected polymers

Comp. No.	^{1}H-NMR parameters (ppm) δ-H
poly (vinyl alcohol)	3.2 (t, 2H, -CH2);2.5 (m, 1H, -CH), 10.1 (OH)



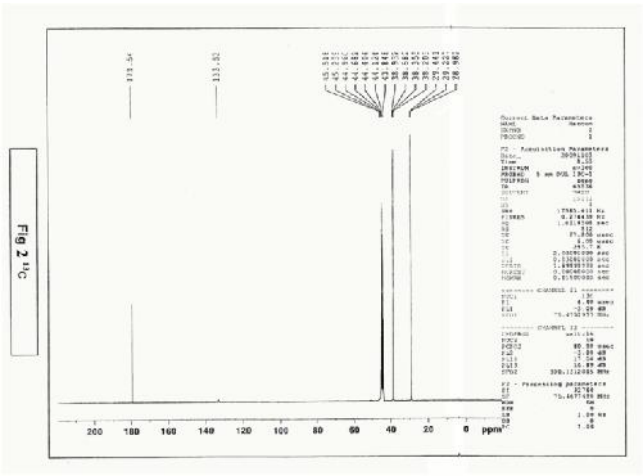


Fig 2 ^{13}C

4. References

[1] Chiellini E.; Corti A.; D'Antone S. ; Solaro R. Prog. Polym. Sci. **2003,** 28, 963.

[2] Finch C.A. Polyvinyl alcohol Developments. New York: Wiley; 1992.

[3] Yasukawa M. D. T.; Kimura H.; Tabata Y.; Ogura Y. Adv. Drug Deliv. Rev. **2001,** 52, 25.

[4] De Prisco N.; Immirzi B.; Malinconico M.; Mormile P.; Petti L.; Gatta G. J. Appl. Polym. Sci. **2002,** 86, 622.

[5] Lozinsky V.I.; Plieva F. M. Enzyme. Microb. Technol. **1998,** 23, 227.

[6] Finley J. H. Anal. Chem. 1961, 33, 1925.

[7] Vieira M.; Tavares C. R.; Bergamasco R.; Petrus J. C. C. J. Membr. Sci. **2001**, 194,273.

[8] Xiao S.; Huang R. Y. M.; Feng X. J. Mem. Sci. **2006**, 286, 245.

[9] Gimenez V.; Mantecon A.; Ronda J. C.; Cadiz V. J. Appl. Polym. Sci. **1997**, 65, 1643.

[10] Gimenez V.; Reina J. A.; Mantecon, A.; Cadiz V. J. Appl. Polym. Sci. **1999**, 40, 2759.

[11] Zhai M.; Yoshii F.; Kume T.; Hashim K. Carbohydr. Polym. **2002**, 50, 295.

[12] Miranda T. M.; Goncalves A. R.; Amorim M.P. Polym. Int. **2001**, 50, 1068- 72.

[13] Gohil J. M., Bhattacharya A.; Ray P. J. Polym. Res. **2006**, 13, 161.

[14] Gebben B.; Hans W. A.; VandenBerg H. W. A.; Bargeman D.; Smolders C. A. Polymer **1985**, 26, 1737.

[15] Macho V.; Fabini M.; Rusina M.; Bobula S.; Harustiak M. Polymer, **l994**, 35, 5773.

[16] Cha W. I.; Hyon S. H.; Ikada Y. Makromol. Chem. **1993**, 194, 2433-41.

[17] McKenna G. B.; Horkay F. Polymer **1994**, 35, 5737.

[18] Kurihara S.; Sakamaki S.; Mogi S.; Ogata T.; Nonaka T. Polymer **1996**, 37, 1123.

[19] Smets G.; Petit B. Makromol. Chem. **1959**, 33, 41.

[20] Kim K. J.; Lee S. B.; Han N. W. Polym. J. **1993**, 25, 1295.

[21] Hansen E. W.; Holm H.; Jahr D. M.; Olafsen J. K.; Stori A Polymer **l997**, 38, 4863.

[22] Zhang H. Z.; Liu B. L.; Luo R.; Wu Y. Z.; Lei D. S. Polym. Degrad. Stab

[23] Bell, VL., Polmer" J.poly.Sci., Vd. **1966**, 13,5.

[24] Encyclopedia of polymer science and Technology, **1969,** Vol.ll.PP-62.

[25] Rodriguez, F. principle of polmer system" 2nd. Ed. McGraw- Hill bookcompany, **1983**.

[26] Ogala Y. and A. Kawasaki, " Mechanistic" aspects of the strecker aminonitrile synthesis". J. chem. Soc., **1971** (13), 325- 329.

[27] Stanley J.W., Beasley and J.G., and Mathison 1. " Eviodence for cationic imine intermediate in N,N - disubstituted - aminonitrile formation", J. Org.chem., **1972**, 37,23, 3746-3748.