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Synthesis of Biologically Potent α-aminophosphonates Derivatives by Nano-catalyst

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ABSTRACT

 α -Aminophosphonate and their derivatives are biologically potent and have received considerable attention in a recent research scenario. The main reason is that they show triguing biological activity. α -Aminophosphonate derivatives are gaining a lot of importance in medicinal chemistry due to their application as enzyme inhibitors, herbicides, antibiotics, pharmaceutical agents and inhibitors of Excitatory Post-Synaptic Potential(EPSP) synthesis, and HIV Protease. It is also important in ati-cancer, anti-HIV, antithrombotic and antibacterial, antioxidant activity. Unfortunately, these compounds have certain limitation such as extraction, purification, of bioactive molecule and their minimum yields. For this reason, many scientists have been orienting their research towards the synthesis of molecules as a new tool to overcome this problems he prime focus of this work is the combination of three reactant derivative of benzaldehyde derivative of aniline, and diethyl phosphonate to form α -aminophosphonates derivatives by multicomponent reaction(KFR). The novel nano-catalyst i.e. polyanilinedoped with manganese (PAni-Mn) was prepared. The catalyst shows excellent catalytic activity, high yields, short reaction times, easy synthesis. The PAni was fully characterized by X-ray diffraction, TEM, SEM, and FT-IR technique.

Keyword: α-Aminophosphonates, EPSP, (PAni-MN), TEM, SEM, FT-IR.

INTRODUCTION

 α -Aminophosphonate is a valuable part of organo-phosphorus compounds due to their similarity in structure and properties to α -amino acids¹. it plays an important role in several field including organic synthesis and various potential applications². Nowadays researchers have been attentively move towards pesticide, biochemistryand medicinal chemistry last few years because they show biological activity. Some α -aminophosphonate show activity against tumor³, activity against microbes⁴, they inhibits the enzyme⁵, they act an antiviral agent⁶, some of the α -aminophosphonates containing alkoxyethyl moieties shows antiviral bioactivity⁷. The Kabachnik-Field reaction and the Pudovic reaction are the two major routs to synthesizing the biologically

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potent α -Aminophosphonates. In the first reaction (Phospha-Manich) it contains three component condensation including aldehyde or ketone, a mine and diethyl phosphate⁸⁻⁹. In the second, it contains imines with >P(O)H reagent¹⁰. The classical version of the "Phospha-Manich" reaction was discovered by independently Kabachnik and fields more than sixty years ago¹¹⁻¹².

The researchers, while synthesizing of α-aminophosphonates used catalyst i.e. efficient Amberlight IRC-74813. An Extremely Efficient Three-Component (KFR) using oxidizing agent Magnesium Perchlorate¹⁴, Zirconium(IV) compounds¹⁵, The Efficient catalyst NbCl5¹⁶, The efficient anthem sulphuric acid¹⁷, Promiscuous Lipase catalyzed (NiSO₄.6H₂O) a new P-C bond formation in (MCR) [18]Tin(II) compound as catalyst for (KFR)¹⁹.

The derivatives of α -Aminophosphonates synthesized by Multicomponent condensation through Kabachnik-Field Reaction¹², are widely explained with a variety of catalysts. Now we have recently reporte the nano (PAni-Mn) catalyst as a novel catalyst used to form α -Aminophosphonates. The (PAni-Mn) Nano catalyst was used for the first time in Kabachnik-Field Reaction for a-Aminophosphonates synthesis. During the last decade, Polyaniline had great importance in the catalytic field²⁰⁻²¹. The doping of the polyanilinewith metal increases the catalytical activity²². The Fe-polyaniline composite Nano-fiber catalystfor chemo selective hydrolysis ofoxime²³. In proposed work first prepares polyaniline, doping should be done with the help of MnCl₂. The synthesized Nano material i.e the nano catalyst (PAni-Mn) is utilized for preparation of a-aminophosphonates derivatives.KFR involes condensation of primary or secondary amines, carbonyl compounds i.e. aldehyde or ketonesand dialkyl phosphite²⁴. The Nano catalyst gives a high yield, short reaction time, it provides high surface area, increased catalytical activity. The synthesized Nano-catalyst was fully characterized by X-ray Diffraction, HR-TEM, FEG-SEM, FTIR.

MATERIALS AND METHODS

Materials

All chemicals are used in these experiments, which are supplied by Sigma Aldrich with high purity.

Synthesis of polyaniline

The chemical oxidation methods were used for PANI-ES synthesis lower than (5°C). mL, Aniline (mL) was dissolved in Hydrochloric acid (70 mL, 1.5 M) his mixture is kept in an ice bath to maintain the temperature below 4-5 °C. The 10 g Oxidizing agent Ammonium Per Sulfate (APS) was dissolved in deionized water. The solution of APS was added drop by drop into monomer solution. This mixture was stirred with a magnetic stirrer up to 4-5 hours²⁵. The polymerization process is carried out, at the end of the polymerization reaction, the green color Polyaniline was formed, washed 2-3 times with D.W. and methanol. Finally, the dark-green composite powder is dried at 70°C in a hot air oven, for 10-12 hours. The final product was grounded to form a green powder (Figure 1).



Fig. 1. Structure of Polyaniline

Preparation of Polyaniline Nano-catalyst

After formation of polyaniline Emeraldine salt (ES), the accurate amount of solution of manganese chloride MnCl₂ slowly and carefully dissolved in polyaniline. The polyaniline manganese chloride solution was kept for stirring with the help of a round bottom flask and Magnetic stirrer (700 RPM) for about 5 hours. After filtration, the product washed 3 times with deionized water and three times with ethanol. The prepared nano catalyst was kept in a hot air oven for 6 h at 70-80°C. In this method the nano particles of Mn was uniformly distributed in polyaniline²⁶⁻²⁷. There is formation of a nano catalyst having a dark green color (Figure 2).



Fig. 2. Freshly PAni-Mn Nano catalyst prepared

RESULT AND DISCUSSION

Polyaniline (X-RD) Analysis

The X-RD technique is used to determine the crystalline nature of polyaniline. ThePANi-ES

gives three different peaks at room temperature i.e. 20.1, 25.3, 26.7°C, respectively as shown in Fig. 2. Polymer is semi-crystalline in nature as the pattern shows sharp peaks due to the presence of Benzenoid and qunonoid groups in the polyaniline28. The sharp peak is observed in the XRD spectrum 2θ =25.2550 Thei nterplanar distance value obtained is 3.35A0. Hence the average crystallite size is calculated on the basis of the Debye Scherer Equation. (D= $k\lambda/\beta$ cos θ) in this equation 1) D=average size of crystallite 2) k=0.89 (Shape of factor), λ =(1.54A0), β =full width at half maximum; θ =angle of diffraction²⁹.

Table 1: Polyaniline data of XRD



The average crystallite size value obtained is 1.387 nm on the basis of XRD data given in Table 1.

SEM Characterization

The main objective of scanning electron microscopy is to determine morphological features and surface characteristics of the compounds. The instruments used JEOL JSM-7600F FEG-SEM. Morphology of polyaniline (ES) shows fibrous in nature particle size is around 1μ m, 100μ m.This shows that the material is in good shape having high surface area, nano fibre which is used for further application. at high temperature polyaniline (ES), tends to formnano-rod like structure. The factors such as polymerization process, polymerization rate, growth of polymers and solvent interfacial tension are also involved in the formation of nano rods³⁰.

TEM (300kV) Characterization

The TEM300kV.can be used to study electron beams to image a Nano particle and generate highly magnified images. The Nano structure of the

polyaniline is shown in the following micrographs on the basis of TEM analysis, the particle size of Polyaniline is very small, i.e.1um, 200nm, 50nm. It is spherical in shapes, having the rough surface.







Fig. 6c. SEM-Pani(Es) 1µm



Fig. 7. TEM Images of PAniFig. 8. TEM Images of PAni(1µm)(200nm)



Fig. 9. TEM Images of PAni 50nm

Fourier- transforms infrared spectroscopy (FTIR)

The prepared polyaniline was identified by FT-IR spectroscopy. The main characteristics peaks observed as 377, 3464, 3232, 2923, 2852, 1663, 1558, 1469, 1299, 1240, 1113, 1006, 878, 797, 679, 562, and 504 cm⁻¹ sample was run in the wavelength 4000-900 cm⁻¹. The FT-IR spectra of synthesized pure polyaniline (ES) is presented in Fig. 9. In a spectrum, the characteristic band observed at 3464-3727 cm⁻¹ as a result of nitrogenhydrogen stretching. The polymers peak observed on 3232, 2923, 2852 cm⁻¹ as a result of asymmetric, symmetric carbon-hydrogen vibration. The C=C of aromatic ring Absorption spectra observed on 1663 cm⁻¹³¹. absorption spectra observed on sharp 1557

cm⁻¹³¹. absorption spectra observed on sharp 1557 cm⁻¹ is the result of C-H stretching in an aromatic compound. The IR spectrum band observed at 1468.59 cm⁻¹ corresponds to C=N stretching in ring aromatic compound. 1240-1299 cm⁻¹. The polymer absorption band of C-N stretching On the basis of this, it confirm the presence of amine group³².

An FT-IR spectrum valued at 1113 cm⁻¹ reveals the C-H bending vibrations. The absorption band lies below 504, 562, 679, 798, 878, 1006, 1044 cm⁻¹ show these spectral values showing the benzene ring being substituted by another group Consequently it shows polymerization³³. The coupling of the phenyl nuclei within the amine group is mainly attached to Para position. On the basis of above FT-IR analysis data confirm that the prepared compound is polyaniline.



Fig. 10. FT-IR spectrum of polyaniline(ES)

Preparation of α -Aminophosphonates using Nano-Catalyst

 α -Aminophosphonate derivates are synthesized through Kabachnik-Fields reaction. Equimolar quantity of aldehyde (10 mmol), different aromatic amine (10mmol), diethyl phosphate (10mmol). Using a catalytic quantity of (PAni-Mn) nano catalyst. In a solvent free environment, they were agitated at room temperature³⁴. The completion of reaction as indicated by TLC³⁵⁻³⁶. The reaction mixture was extracted with ethyl acetate and quenched with water (10 mL). The formation of pure α -aminophosphonate follows the purification of the compound in silica gel.

RESULT AND DISCUSSION

Following α -Aminophosphonate derivatives were prepared.

Diethyl-3-chlorophenylamino-4-hydroxy-3methoxy-5-nitrophenyl methylphosphonates.

M.F. $C_{18}H_{22}CIN_2O_7P$ M.W= 444, dark brown color m. p. = 177-179°C, yield=88% ¹HNMR(300MHz, DMSO-*d*₀) δ_{H} ; 10.3 (s, 1H,-OH), 8.90-6.58 (m, 6H, Ar-H), 5.02-xx (m, 1H,N-H), 4.05-xx (m, 1H,P-CH), 3.81 (q, 4H, P-OCH2), 3.15 (s, 3H, -OCH₃), and 1.12 (s, 3H, -OCH₃) (t, H, -OCCH₃)./1162 MHz, 32 M/Z=444 and 446 with a 3:1 ratio for 31P-NMR37.



Diethyl (4-methoxyphenyl)-N -(Phenyl amino) methylphosphonate

M.F. $C_{18}H_{24}NO_4P$ M.W=349, dark yellow color m.p. = 57-59°C, yield=92% ¹HNMR(300MHz, CDCI3) δ_{H} ; 1.01-1.07(m,3H), 1.17-1.21(m, 3H), 3.71 (s,3H) 3.62-3.64, 3.84-3.88, 4.04-xx(m, 4H), 4.57-4.68 (m, 2H), 6.51-6.62(m, 3H), 6.76-6.80(m, 2H) and 7.0-xx(m,2H), 7.30-7.32(m, 2H). 31P-NMR (16.9 MHz, DMSO- d_6) With a 3:1 ratio, 30.6 M/Z equals 444 and 44638.



Diethyl (2-chloro phenyl amino) nitrophenyl methyl (4-hydroxy-3-methoxy) phosphonate

 $\begin{array}{l} \text{M.F. } C_{18}\text{H}_{22}\text{CIN}_{2}\text{O7P M.W}{=}408, \text{brown color} \\ \text{m.p.} = 174{\text{-}}177^{\circ}\text{C}, \text{ yield}{=}87\% \ ^{1}\text{HNMR}(300\text{MHz}, \\ \text{DMSO-}\textit{d}_{\textit{\theta}})\delta_{\text{H}}{:} 8.20(\text{s}, 1\text{H}, \text{ArH}), 7.28(\text{d}, 1\text{H}, \textit{J}{=}6.5 \\ \text{Hz}, \text{ArH}), 6.989\text{d}, 1 \text{h}, \textit{J}{=}6.5 \text{Hz}, \text{ArH}), 6.92 \\ (\text{d}, 2\text{H}, \textit{J}{=}6.5\text{Hz}, \text{ArH}) 6.70(\text{s}, 1\text{H}, \text{ArH}), 4.80 \\ (\text{d}, 1\text{H}, \text{JCHPO}{=}23.7 \text{ Hz}, \text{CHP}) 4.02{\text{-}}4.12(\text{m}, 2\text{H}, \\ \text{OCH}_2\text{CH}_3), 3.95{\text{-}}3.98(\text{m}, 1\text{H}, \text{OCH}_2\text{CH}_3), 3.90 \\ (\text{s}, 3\text{H}, \text{OCH}_3)3.70{\text{-}}3.75(\text{m}, 1\text{H}, \text{OCH}_2\text{CH}_3), 1.26 \\ (\text{t}, 3\text{H}, \textit{J}{=}6.4 \text{ Hz}, \text{CH}_3), 1.15(\text{t}, 3\text{h}, \textit{J}{=}6.4 \text{ Hz}, \text{CH}_3) 31\text{P}{\text{-}}\text{NMR} \\ (16.5 \text{ MHz}, \text{DMSO-}\textit{d}_{s}, \text{at } 30.4\text{M}/\text{Z}{=}440 \text{ and } 44239. \\ \end{array}$



Diethyl(4-methoxy phenyl)-N- (4-chlorophenyl amino) methylphosphonates

M.F. $C_{18}H_{23}$ CINO₄P M.W= 366, yellow color m.p. = 161-163°C, yield=91% ¹HNMR(300MHz, DMSO- d_{g}) δ_{H} ; 7.41(d, 2H, *J*=7.4Hz, Ar-H), 7.14 ((t, 2H, *J*=7.4 Hz, ArH,), 6.91(d, 2H, *J*=8.4 Hz, Ar H), 6.71(d, 2H, *J*=8.4 Hz, Ar-H), 6.71(t, 1H, *J*=7.24, Hz, Ar-H), 6.62(d, 2H, *J*=8.1 Hz, Ar-H), 4.76 (s, 1H, NH), 4.75(d, 1H, *J*CHPO=40.0. Hz, CHP), 4.12-4.18(m, 2H, OCH₂CH₃), 3.94-4.0 (m, 1H, OCH₂CH₃), 3.78(s, 3HOCH₃), 3.70-3.76 (m, 1H, OCH₂CH₃), 1.30(t, 3H, *J*=7.2Hz, CH₃), 1.18 (t, 3H, *J*=7.2 Hz, CH₃).³¹P-NMR (16.1 MHz, DMSO- d_{2}) 30.1M/Z=448 and 447 with 3:1 ratio⁴⁰.



Diethyl phenyl (phenyl amino) methyl phosphonate

M.F. $C_{17}H_{22}NO_3P$ M.W=318, white color m.p. =93-95°C, yield=89% ¹HNMR(300MHz, CDCl₃) δ_{H} ; 1.1(3H, JHH= 7.1Hz, t, OCH₂CH₃); 1.3 (3H, JHH=7.1Hz, t, OCH₂CH₃); 3.74-4.4 (m, 4H, OCH₂ CH₃); 5.1(s, 1H, NH); 4.8(1H, JHP=24.6 Hz, d, CHP); 5.1(m, 10H, Ar-H), 31P –NMR (16.3 MHz DMSO- d_{θ}) with 3:1 ratio.⁴¹



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CONCLUSION

Using the one-pot, three-component Kabachnik-field reaction, it was possible to synthesize novel derivatives of α -aminophosphonates. The use of different types of aldehyde, substituted aniline and dialkyl phosphate under solvent free condition using a novel nano-catalyst (PAni-Mn). The nano catalyst doped polyaniline with manganese (PAni-Mn) has greater efficiency, simple reaction condition, easy to handle and efficient. The Nano-catalyst was characterized by X-ray diffraction, HR-TEM, FEG-SEM, FTIR technique. All the prepared compounds were analyzed. It is worth mentioning that this catalyst is first used in the synthesis of α -aminophosphonates.

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