Int. J. Curr. Res. Chem. Pharma. Sci. 2(9): (2015):66–70

INTERNATIONAL JOURNAL OF CURRENT RESEARCH IN CHEMISTRY AND PHARMACEUTICAL SCIENCES

(p-ISSN: 2348-5213: e-ISSN: 2348-5221)
www.ijcrcps.com

Research Article

SYNTHESIS OF SALICYLIC ACID –FORMALDEHYDE POLYMERS

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Abstract

Abstract Details of only typical methods are furnished. In other cases only the amount of the reactants used is given. Condensation of salicylic acid (0.02 Mole) with formaldehyde (0.016 Mole) in presence of aqueous 40% H_2SO_4.

Keywords: Water bath, Thermometer, Spectrophotometer, Condensation.

Introduction

The polymer sample obtained by each of above methods was light brown in colour. It was a light weight powder. The solubility behavior of all the polymer samples have been studied. The salicylic acid – formaldehyde polymer is soluble in acetone while p-hydroxy benzoic acid – formaldehyde solution is not completely soluble in acetone. The polymer samples were found to be completely soluble in other solvents like, ethanol, Dioxane, tetrahydrofuran, Di methyl formamide and di methyl sulphoxide. All the solvents gave reddish brown colored polymer solutions.

The polymer samples are soluble in aqueous alkali. These polymer samples dissolve in aqueous bicarbonate liberating carbon dioxide. The solution of the sodium salt of polymer in water affords violet-blue precipitates with aqueous ferric chloride solution. From the solution in aqueous alkali the product almost completely separated on acidification.

Materials and Methods

1. Formation of salicylic acid formaldehyde polymer sample .

A mixture of salicylic acid (0.25 Mole), 37% formaldehyde solution (0.25 Mole) and 120 ml 40% sulfuric acid was refluxed with good stirring at 125-130^{0}C for 6 hours. During this time, the solid polymer was formed. The solid was filtered, washed with a large amount of water and then with boiling water. The air-dried polymer sample was sox let-extracted with benzene to remove unreacted monomer. The polymer sample was dissolved in 1:1 mixture of acetone and ethanol and reprecipitated as a pasty mass by adding distilled water. The pasty mass was washed by decantation and collected. The mass was dissolved in dilute alkali and reprecipitated by gradual addition of 1:1 conc. HCl – water mixture with stirring. The polymer sample did not melt up to 360^{0}C. The yield was 36.0 gm. About 10 gm polymer sample was fractionated.

Analysis	C(%)	H(%)
Found	63.50	4.00
Required for repeat unit $C_8H_6O_3$	64.00	4.00

2. Formation of 4-hydroxy benzoic acid polymer sample

A mixture of 4-hydroxy benzoic acid (0.25 Mole), 37% formaldehyde solution (0.25 Mole) and 500 ml 40% sulfuric acid was refluxed with good stirring at 125-130^{0}C for 6 hours. During this time, the solid polymer was formed. The solid was filtered, washed with a large amount of water and then with boiling water. The air-dried polymer sample was Sox let-extracted with benzene to remove unreacted monomer. The polymer sample was dissolved in 1:1 mixture of acetone and ethanol and reprecipitated as a pasty mass by adding distilled water. The pasty mass was washed by decantation and collected. The mass was dissolved in dilute alkali and reprecipitated by gradual addition of 1:1 conc. HCl – water mixture with stirring. The polymer sample did not melt up to 360^{0}C. The yield was 35.0 gm. About 10 gm polymer sample was fractionated.

Analysis	C(%)	H(%)
Found	64.04	4.05
Required for repeat unit $C_8H_6O_3$	64.00	4.00

3. Formation of Resorcylic acid - formaldehyde polymer sample

A mixture of Resorcylic acid (0.25 mole), 37% formaldehyde solution (0.25 mole) and 500 ml 40% sulfuric acid was refluxed with good stirring at 125-130^{0}C for 6 hours. During this time, the solid polymer was formed. The solid was filtered, washed with a large amount of water and then with boiling water. The air-dried polymer sample was soxhlet-extracted with benzene to remove unreacted monomer. The polymer sample was dissolved in 1:1 mixture of acetone and ethanol and reprecipitated as a pasty mass by adding distilled water. The pasty mass was washed by decantation and collected. The mass was dissolved in dilute alkali and reprecipitated by gradual addition of 1:1 conc. HCl – water mixture with stirring. The polymer sample did not melt upto 360^{0}C. The yield was 35.0 gm. About 10 gm polymer sample was fractionated.

Analysis	C(%)	H(%)
Found	57.81	3.61
Required for repeat unit $C_8H_6O_4$	57.83	3.61

Table 2.1. Infrared spectral characteristics general.

OH deformation	**950-900 cm^{-1}**
-CH_2- Bridge	
CH stretching	2870-2845 cm^{-1}
CH bending	1465 $\pm$ 20 cm^{-1}
Aromatic CH	
C-H stretching	3030 cm^{-1}
C-H bending	1225 – 950 cm^{-1}
C=O vibration	
Internal hydrogen bonding	1680-1650 cm^{-1}

Results and Discussion

The spectra of the polymers described in this chapter have been taken in KBr pellet. Carefully purified KBr was used for the purpose. Absence of KBr was checked by examining IR spectrum of pure KBr pellet. The pellet for the IR study was prepared by mixing 2.0 mg of polymer sample with 1.0 gm of pure KBr in a mini-ball mill and compressing this mixture in a disc at a pressure of 20000 psi under vaccum. The disc thus prepared was transparent and very lightly colored. The spectrum of pellet was taken on IR-5 spectrophotometer. IR spectrum of salicylic acid – formaldehyde, 4-hydroxy benzoic acid – formaldehyde and Resorcilic acid – formaldehyde polymers samples prepared from them have been taken by this method. The positions of all bands have been noted and presented in tables 2.2 and 2.3. The spectra are presented in figure 2.1 to 2.3.

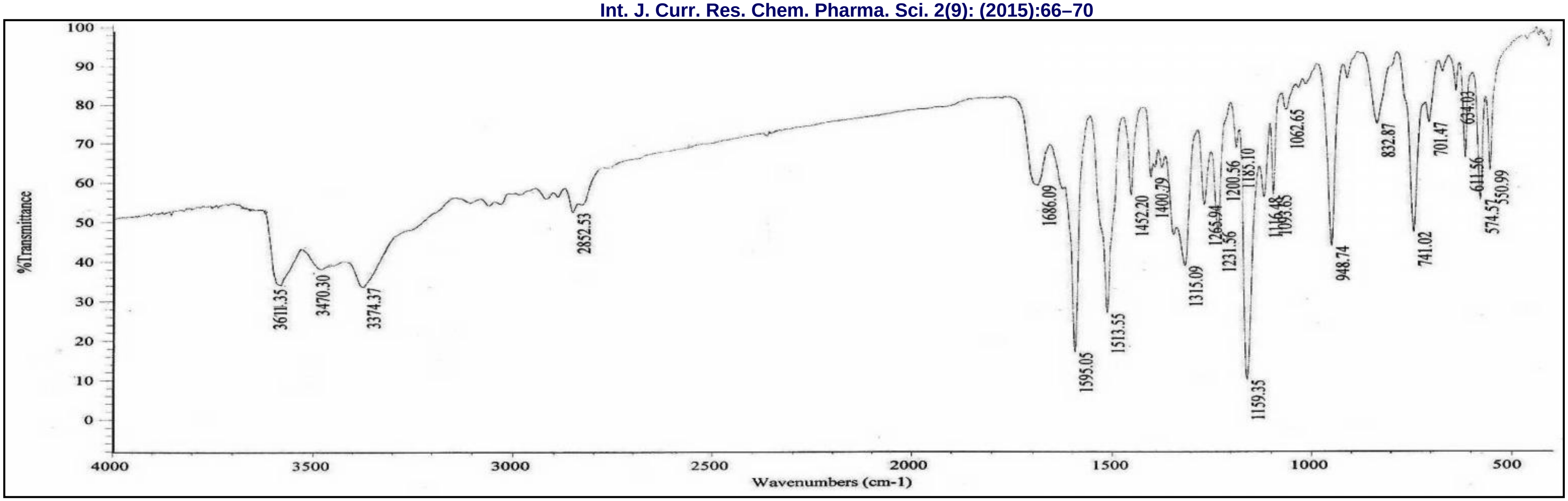

Figure 2.1. IR spectrum of salicylic acid – formaldehyde, 4-hydroxy benzoic acid – formaldehyde and Resorcilic acid – formaldehyde polymers

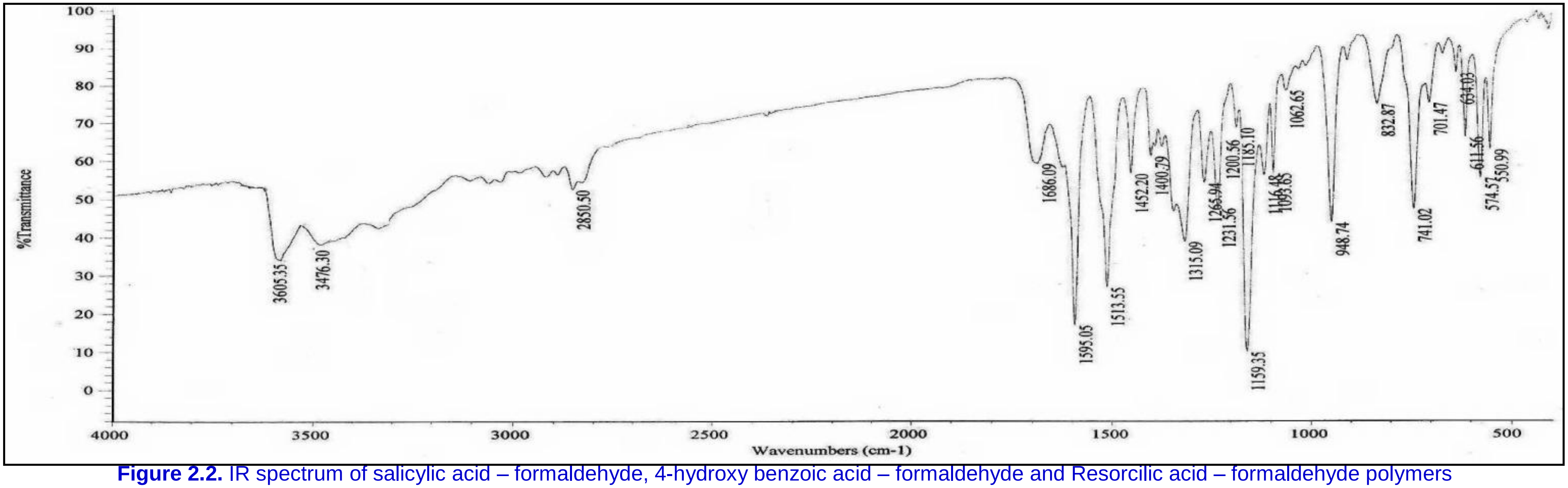

Figure 2.2. IR spectrum of salicylic acid – formaldehyde, 4-hydroxy benzoic acid – formaldehyde and Resorcilic acid – formaldehyde polymers

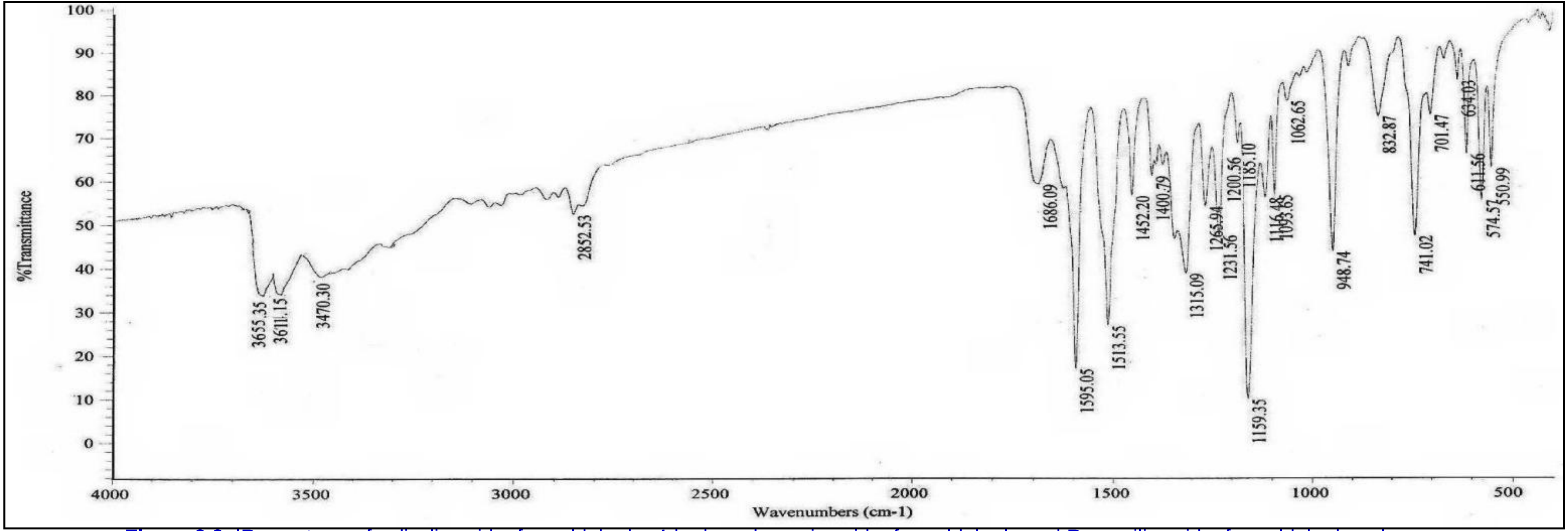

Figure 2.3. IR spectrum of salicylic acid – formaldehyde, 4-hydroxy benzoic acid – formaldehyde and Resorcilic acid – formaldehyde polymers

Table 2.2 Anticipated spectral features of prepared oligomers

Sr. No.	Group	Salicylic acid - Formaldehyde Oligomer cm^{-1}	p-OH Benzoic acid-Formaldehyde Oligomer cm^{-1}	Resorcilic acid-formaldehyde oligomer cm^{-1}
1	Phenolic -OH stretching (Free)	3611.35	3605.35	3655.35, 3611.15
2	Carboxylate -OH stretching (Free)	3470.30	3476.30	3470.30
3	O-H bending (in plane)	1200.56	1200.56	1200.56
4	C-O stretching	1315.09	1315.09	1315.09
5	OH deformation	948.74	948.74	948.74
6	CH stretching	2852.53	2850.50	2852.53
7	CH bending	1452.20	1452.20	1452.20
8	Aromatic C-H stretching	3374.37	3374.35	3374.30
9	Aromatic C-H bending	1159.35	1159.35	1159.35
10	C=O stretching	1686.09	1686.09	1686.09

Conclusion

The examination of all the three IR spectra of three type of polymers revels that:

1. All the spectra are Identical in all the aspects.
2. The bond from 3600 to 2500 with the inflexion due to –OH of -COOH and phenolic -OH group.
3. The inflexion at 2850 and 2920 cm^{-1} are due to CH_2 bridge.
4. The strong band at 1680 cm^{-1} is due to –COOH group.
5. Other bands are appeared at their respectable position

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