

# Characterization of Phenol-Formaldehyde Prepolymer Resins by In Line FT-IR Spectroscopy<sup>†</sup>

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## Abstract

Different resol phenol-formaldehyde prepolymer resins have been synthesized with different formaldehyde / phenol ratios. The phenolic resin composition depends on monomer ratio, catalyst, reaction conditions, and residual free monomers. Temperature and pH conditions under which reactions of phenols with formaldehyde are carried out have a profound effect on the characteristics of the resulting products. Three reaction sequences must be considered: formaldehyde addition to phenol, chain growth or prepolymer formation and finally the crosslinking or curing reaction. Two prepolymer types are obtained depending on pH, novolacs in an acidic pH region whereas resols by alkaline reaction. Resol resins are synthesized with a molar excess of formaldehyde ( $1 < F/P < 3$ ). These are mono- or polynuclear hydroxymethylphenols which are stable at room temperatures, but are transformed into three dimensional, cross linked, insoluble and infusible polymers by the application of heat.

An ATR-FTIR spectrometry technique (ReactIR 4000) with light conduit and diamond-composite sensor was used to perform in-line monitoring of phenol-formaldehyde prepolymer synthesis. This technique was found to be ideal for determining residual free phenol and formaldehyde, individual phenol and formaldehyde conversions and prepolymer composition changes as a function of time when the condensation reaction was carried out. The kinetics data obtained through the ReactIR 4000 in-line reaction analysis system agreed well with those determined by the traditional titration method. ReactIR technology replaces time consuming and inaccurate off-line methodology.

**Key words:** phenol-formaldehyde resin, in-line IR spectroscopy

## Introduction

Since their introduction in 1910 the relatively inexpensive and highly versatile family of phenolic resins has played a vital role in construction, automotive, electrical and appliance industries.<sup>1,2,3</sup> Temperature and pH conditions under which reactions of phenols with formaldehyde are carried out have a profound effect on the characteristics of the resulting products. Three reaction sequences must be considered: formaldehyde addition to phenol, chain growth or prepolymer formation and finally the crosslinking or curing reaction.<sup>4-8</sup> Two prepolymer types are obtained depending on pH, novolacs in an acidic pH region whereas resols by alkaline reaction. Resol resins are synthesized with a molar excess of formaldehyde ( $1 < F/P < 3$ ). These are mono- or polynuclear hydroxymethylphenols which are stable at room temperatures, but are transformed into three dimensional, crosslinked, insoluble and infusible polymers by the application of heat. Recently, urea has been introduced into phenol-formaldehyde (PF) resins

to improve their curing, to lower the content of free formaldehyde and to reduce the cost of the resin.<sup>9,10</sup> To clarify the complicated relationship between the phenol ring and amino functional group our research work started with the study of phenol-formaldehyde resins.

The aim of our work was to study the PF resin preparation process. The influence of molar ratio between phenol and formaldehyde on the PF synthesis and on the composition of the synthesized product was investigated. Furthermore, the pH value of the reaction mixture was varied to study their influence on the structure of PF resin. An ATR-FTIR spectrometry was found to be a successful and informative analytical tool for determining individual phenol and formaldehyde conversions and prepolymer composition changes as a function of time when the condensation reaction was carried out.<sup>11,12,13</sup> We detected the different bridges formations during the reaction of two hydroxymethyl groups under alkaline conditions. According to review of the literature available to us, the in-line ATR-FTIR technique has not been applied in the studies of PF

**Table 1.** Reaction conditions of phenol-formaldehyde resin synthesis, concentrations of phenol and formaldehyde at  $t=0$  and free formaldehyde in the resin at the end of reaction synthesis.

Synthesis	Molar ratio Phenol:Formaldehyde	$C_{A0}$ Phenol mol/L	$C_{A0}$ Form. mol/L	$T^*$ , °C	t, h	pH	% Free Form.
Synthesis 1	1:1.6	4.05	6.22	61,5	3	8	3.35%
Synthesis 2	1:1.8	3.78	6.58	55	3	8	2.32%
Synthesis 3	1:1.8	3.78	6.58	21,3	3	7	8.86%
Synthesis 4	1:1.8	3.78	6.58	62	3	8	2.77%
Synthesis 5	1:1.9	3.68	6.5	65	3	8	5.77%
Synthesis 6	1:2	3.57	6.90	70	3	8	3.75%

\* Temperature at which we started to collect mid FTIR spectra of the condensation components.

resins synthesis. Different resol prepolymer resins have been synthesized with different F/P ratios. An important structural factor determined quantitatively for resol prepolymer resins is the F/P ratio after the reaction. In order to evaluate the ability of the ATR-FTIR spectroscopic method, the kinetic data obtained through the IR in-line analysis were compared to results from the traditional titration method.<sup>14</sup>

## Experimental

### Materials

Phenol (99+ %), 37% formaldehyde aqueous solution and sodium hydroxide (98%) were used. All the chemicals were supplied by Aldrich.

### Resin synthesis

Different resol prepolymer resins (Table 1) were prepared with the same amount of catalyst (NaOH) but with different F/P ratios: R1, R2, R3 and R4 with F/P ratios of 1.6, 1.8, 1.9 and 2.0, respectively. PF resin was synthesized in a 500 ml batch reactor fitted with a reflux condenser, a mechanical stirrer, a digital thermometer and an IR probe. Several batch condensations of phenol and formaldehyde were conducted at 90 °C. Reaction vessels were heated to the same reaction temperature. The recipes used are shown in Table 1.

All resins were prepared by reacting the required amounts of phenol (80% solution in water) with varying amounts of formaldehyde (37% solution in water, Merck) in the presence of the catalyst. The resulting reaction mixture was stirred and heated to 90–95 °C until the mixture became cloudy. Afterwards, the resin is used as it is or it is neutralized. In the first set of experiments the reproducibility of the resin synthesis was investigated to determine the amounts of free phenol and free formaldehyde.

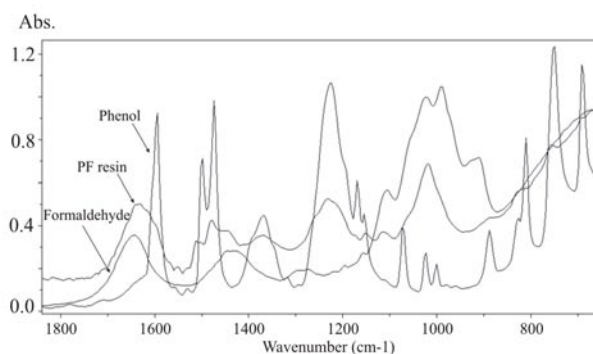
### FTIR spectroscopy

A ReactIR™ 4000 reaction analysis system equipped with a light conduit and DiComp (diamond

composite) insertion probe was used to collect mid-FTIR spectra of the condensation components. IR spectra were collected every three minutes in the wavenumber range between 4000 and 650  $\text{cm}^{-1}$  at resolution 8  $\text{cm}^{-1}$ . These spectra were used to calculate the amounts of free phenol and free formaldehyde and to some extent the PF resin composition. At the end of the synthesis the traditional titration method for determining free formaldehyde was carried out as well as residual phenol content was determined by gas chromatography.<sup>15</sup>

## Results and discussion

ATR-FTIR spectrometry was utilized to monitor the condensation reaction in real time. In-line data acquisition was performed by immersing the transmission probe directly into the reactor. Assignment of the characteristic peaks of phenol and formaldehyde was done using the previously collected spectra of both reactants in aqueous solution. The spectra of phenol and formaldehyde solutions are presented in Figure 1 and collected in Table 2. The assignment of characteristic peaks was done on the basis of relevant literature.<sup>16,17</sup> In Figure 1 a typical IR spectrum of a phenol-formaldehyde resin (synthesis 2) is also represented.



**Figure 1.** FTIR spectra at the wavelengths in the range of interest for phenol, formaldehyde and phenol-formaldehyde resin type resol (synthesis 2, no.61).

**Table 2.** Functional groups and observed wavenumbers for phenol and formaldehyde and their literature values.

Literature data of wavenumber, $\text{cm}^{-1}$	Observed wavenumber, $\text{cm}^{-1}$	Functional group
Phenol		
3400	3308	OH stretch
/	3026	CH unsaturated stretch
1605	1594	C = C aromatic ring
1510	1498	C = C aromatic ring
1485	1475	C = C aromatic ring
1380	1370	OH in-plane bend
1240	1224	C-C-O asymmetric stretch
1180	1170	CH aromatic, in-plane
1160	1153	C-O stretch
1060	1073	single bond C-O stretching vibrations of -C-OH group
1020	1023	-C-OH
990	999.7	-C-H
880	887.8	-C-H
800	810.6	asymmetric stretch of phenolic C-C-OH
747	748.8	CH aromatic, out-of-plane
680	690.9	ring bend
Formaldehyde		
3400	3350	OH stretch
2822	2980	CH stretch, general
2724	2914	CH bend overtone
1722	1644*	C = O stretch (overlapped with OH scissors of water)
1390	1447	C-H bend
/	1158	C-O stretch
/	1108	-C-OH
/	1023	-C-OH
/	992	C-H
/	911	
/	671	

**Table 3.** Functional groups and observed wavenumbers for phenol-formaldehyde resin type resol and their literature values.

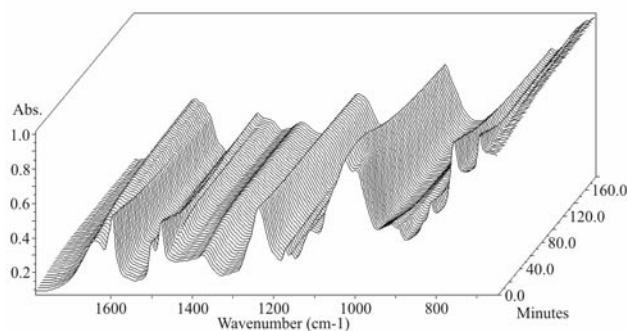
Literature data of wavenumber, $\text{cm}^{-1}$	Observed wavenumber, $\text{cm}^{-1}$	Functional group
3400	3389	OH
2925	2953	in phase stretching vibration of -CH <sub>2</sub> - alkane
2850	2895	out of phase stretching vibration of -CH <sub>2</sub> - alkane
	1633	C = C aromatic ring
1610	1610*	C = C aromatic ring
1517	1552	C = C aromatic ring
1504	1513	C = C aromatic ring
1480	1478	C-H aliphatic
1460	/	-CH <sub>2</sub> - deformation vibration
1450	1451*	C = C benzene ring obscured by -CH <sub>2</sub> - methylene bridge
1390	1370	OH in plane
1378		
1237	1235	asymmetric stretch of phenolic C-C-OH
1153	1154	C-O stretch
1100	1116	asymmetric stretching vibration of C – O-C aliphatic ether
1045	1058*	single bond C-O stretching vibrations of -CH <sub>2</sub> OH group
1000	1019	aliphatic hydroxyl
976	976*	1,2,4-substituted benzene ring
885	887.8	CH out-of-plane, isolated H
835	826	CH out-of-plane, para-substituted
760	756.6	CH out of plane, ortho-substituted
694	694.8	adjacent 5H

\*observed IR spectral frequencies of 2,4,6-trihydroxymethyl phenol

Characteristic absorbances of the phenolic resin are assigned in Table 3 using the literature.<sup>18,19,20</sup> These signals were helpful in identifying the components of the condensation reaction of phenol and formaldehyde. We do have to emphasize that the literature assigned absorption bands to the various chemical groups did vary, as might have been expected.<sup>19</sup>

The normalized phenol-formaldehyde resin spectra at the wavelengths in the range of interest for the PF resin synthesis are shown in Figure 2. The spectra were recorded at a resolution of  $8 \text{ cm}^{-1}$  with 128 scans for each spectrum. The reaction mixture spectra of PF resin run were collected every 3 minutes for a total of 180 minutes at wavenumbers from  $4000\text{--}650 \text{ cm}^{-1}$ . As shown in Figure 2, the characteristic peaks of phenol at 1594, 1498, 1224, 1170, 999.7, and  $748.8 \text{ cm}^{-1}$  diminished with increasing reaction time, while the absorbance band of hydroxymethyl and methylene groups increased. The

peaks at 1594 and  $1498 \text{ cm}^{-1}$  corresponded to the C=C aromatic ring vibrations. The peaks at 1224 and  $1170 \text{ cm}^{-1}$  corresponded to the C-C-O asymmetric stretch and C-H in plane deformations, respectively, while the 999.7, and  $748.8 \text{ cm}^{-1}$  peaks belonged to the C-H out of plane vibrations. The peak at  $1370 \text{ cm}^{-1}$  which corresponded to the phenol O-H in plane bend (Figures 1 and 2) was also detected for phenol-formaldehyde resin. The width of the band at  $1370 \text{ cm}^{-1}$  indicates it is due to an O-H rather than C-H or C-C bond. The formaldehyde aqueous solution was added into the reactor at the beginning of the process, therefore the characteristic peaks of methylene glycol C-O, O-H, and C-H bends at  $1108 \text{ cm}^{-1}$ ,  $1023 \text{ cm}^{-1}$ , and  $992 \text{ cm}^{-1}$  were observed in the first spectrum, respectively (Figure 2, Table 2). After the reactor content heated to preferred temperature a sharp decrease of phenol and formaldehyde peaks was observed as a result of the reaction.

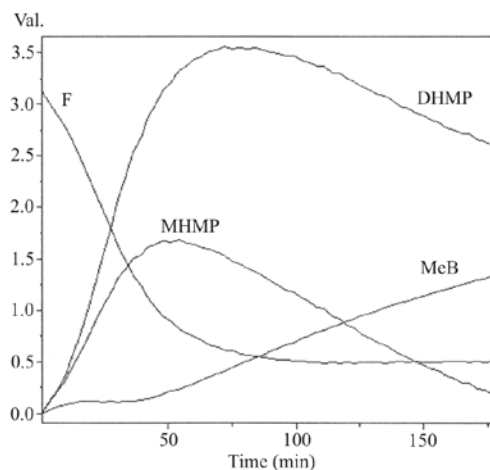


**Figure 2.** Reaction profile shows significant changes as the polymerization of PF resin (molar ratio F/P = 1.8/1) occurs in range of  $1800\text{ cm}^{-1} - 650\text{ cm}^{-1}$ .

At the beginning the characteristic peak of the formaldehyde at  $911\text{ cm}^{-1}$  decreased with increasing reaction time and afterwards slightly increased due to the conversion of methylene ether bridges into methylene bridges with the loss of formaldehyde and water. The absorbances for other characteristic groups were less observable because they overlapped with other absorbances of the product. It can be observed (Figures 2) that an additional peak occurred at  $1610\text{ cm}^{-1}$ . This peak was attributed to C=C band vibrations of the product aromatic ring. In the phenol-formaldehyde resin spectrum additional characteristic signals of methylene bridge C-H bend at  $1478\text{ cm}^{-1}$  and methylene-ether bridge C-O-C bend at  $1116\text{ cm}^{-1}$  were present.

In Figure 3 the concentration profiles for phenol, formaldehyde, and the reaction product were generated by the use of ConcIRT™ Opus I software. The decrease of free formaldehyde and on the other hand, the increase of methylene bridges (MeB) as well as the formation of mono-hydroxymethyl (MHMP) and di-hydroxymethyl phenols (DHMP) was observed from the concentration profiles. The concentration of mono-hydroxymethyl phenols increased and then decreased due to the formation of di-hydroxymethyl phenols and methylene bridges. The reaction rate of the formation of methylene bridges increased when the mono-hydroxymethyl groups reached the maximum. In the first 50 minutes the concentration of hydroxymethyl groups increased and afterwards, when methylene-ether bridges or methylene bridges were formed, the concentration started to decrease. In the same time di-hydroxymethyl groups were also formed. The concentration of di-hydroxymethyl groups increased till the maximum in 75 minutes and then started to decrease due to the formation of methylene bridges. The concentration of formaldehyde began to decrease from the beginning of the experiment. It was estimated that almost all the free formaldehyde reacted in 75 minutes of the reaction, while the methylene and

methylene-ether bridges continued to react till the end of experiment.

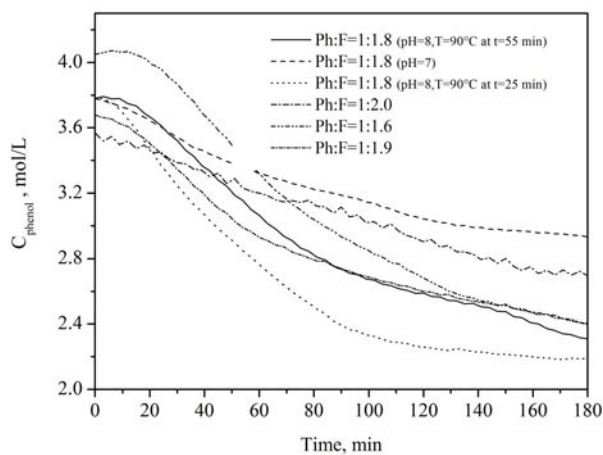


**Figure 3.** The concentration profiles for formaldehyde (F), mono-hydroxymethyl groups (MHMP), di-hydroxymethyl groups (DHMP) and methylene bridges (MeB) during PF synthesis.

Figures 4 and 5 present the time evolution of phenol and formaldehyde consumption determined by means of the in-line IR measurements. At the end of the synthesis the traditional titration method for determining free formaldehyde was carried out as well as residual phenol content was determined by gas chromatography. The calculated residual phenol content of final products was compared with those observed by GC. In spite of the different principle of measurements the agreement between the results is satisfactory, within  $\pm 10\%$  error. The consumption of monomers (phenol and formaldehyde) during the reaction was estimated by calculating the ratio between the absorbance at  $1594\text{ cm}^{-1}$  (phenol) and the absorbance at  $911\text{ cm}^{-1}$  (formaldehyde) at reaction time  $t$  and the corresponding peaks at the start of the polymerization reaction ( $t = 0$ ), according to Equation (1).

$$X (\text{mol}\%) = \left(1 - \frac{\text{peak height at time } t}{\text{peak height at time } t=0}\right) \times 100 \quad (1)$$

The absorbance of the characteristic peak was measured as the peak height referred to a two point baseline. The in-line end values of the formaldehyde consumption (conversion) agreed well with those from the off-line traditional titration method (Table 1). In Table 1 the reaction conditions of phenol-formaldehyde resin synthesis, concentrations of phenol and formaldehyde and free formaldehyde at the end of reaction synthesis are listed. The ATR-FTIR probe also detected the oscillation of the temperature as a change in the ATR-FTIR absorbance peak heights.

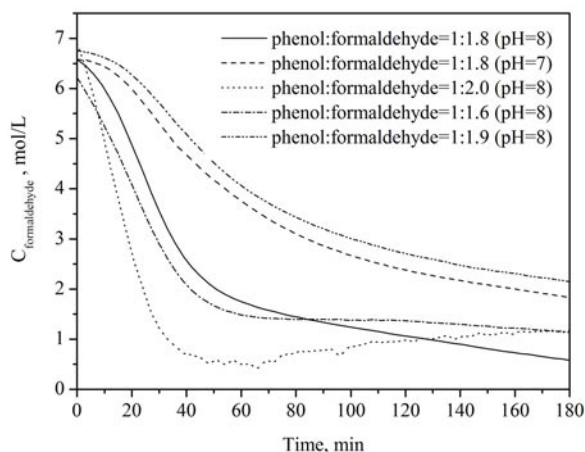


**Figure 4.** Concentration profile of phenol in the synthesis of phenol-formaldehyde resin at different molar ratios formaldehyde : phenol.

The concentration profiles for phenol in the synthesis of phenol-formaldehyde resin at different molar ratios formaldehyde : phenol agreed with the well known theory that in the synthesis of PF resin the pH value of the reaction mixture has a great influence as well as the temperature. For the synthesis of PF resin at pH=7 the decrease of phenol is lower regarding to the synthesis of PF resin at pH=8. At higher temperatures the consumption of phenol is higher.

The weight % of free formaldehyde in the synthesis 3 and 5 are higher due to the lower temperature and lower pH value of the reaction mixture. The influence of the pH value of the reaction mixture prevailed over the influence of the reaction temperature. The lower rate of consumption of formaldehyde in the synthesis 5 (1 : 1.9) can be ascribed to the lower pH value of the reaction mixture due to the higher amount of formaldehyde at the start of the reaction and on the other hand due to the lower starting temperature. The concentration of formaldehyde (Figure 5) in the synthesis of PF resin with the F/P ratio 2/1 decreased for 35 minutes, when it reached the minimum and started to increase due to the formation of methylene bridges from the methylene-ether bridges. In this reaction free formaldehyde is a side product. In order to observe the influence of pH on the synthesis of PF resins the reaction was proceeded at pH=7 and pH=8. From the Figure 5 it can be observed that the concentration of formaldehyde for the reaction condition of pH=7 slowly decreased, regarding to the concentration of formaldehyde for the synthesis at pH=8. The concentration of the catalyst is very important for the reaction rate. For the synthesis of PF resin with a molar ratio of 1.8/1 at pH=8 the concentration of free formaldehyde decreased from 6.58 mol/L to 1.74 mol/L after the first hour and at the end of the experiment the concentration of free

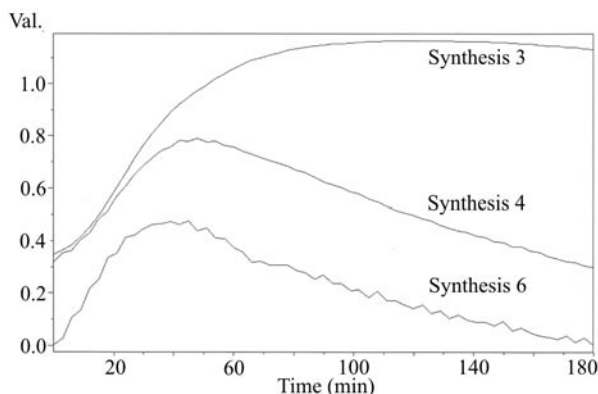
formaldehyde was 0.56 mol/L. The in-line end values of the formaldehyde consumption agreed well with those from the off-line traditional titration method.



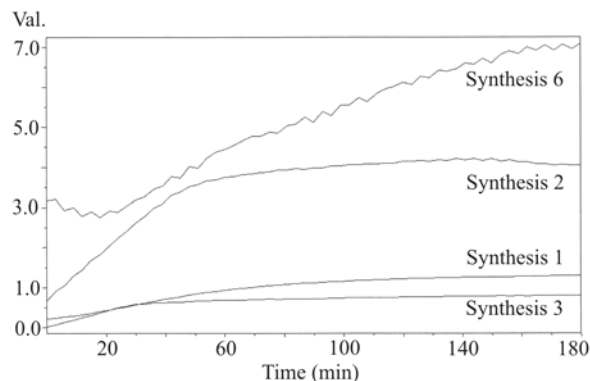
**Figure 5.** Concentration profile of formaldehyde in the synthesis of phenol-formaldehyde resin at different molar ratios formaldehyde : phenol.

The concentration profiles for hydroxymethylphenols and for methylene bridges are shown in Figures 6 and 7, respectively. The profiles were generated by the use of Conc IRT™ Opus I software. The concentration profiles of hydroxymethylphenols in the synthesis of PF resins for different molar ratios of F/P are shown in Figure 6. The reaction rate of hydroxymethylphenols increased with the increasing ratio between F/P. The maximum of hydroxymethylphenol concentration was reached in 38 minutes for the molar ratio of 2/1 and in 48 minutes for the molar ratio of 1.8/1 and then started to decrease due to the formation of methylene and methylene-ether bridges. For the synthesis 3 with the molar ratio of 1.8/1 it is obvious that the reaction rate of hydroxymethylphenol is actually the same but the reaction temperature was too low for the formation of methylene or methylene-ether bridges. The PF resin synthesized under the conditions mentioned was soluble in water which corresponds to the PF resins with a high concentration of hydroxymethyl groups. The polymerization of hydroxymethyl phenols was hindered by lower pH values while the influence of lower starting temperatures was not so significant.

The concentration profiles of methylene bridges in the synthesis of PF resins with different molar ratio between F/P are represented in Figure 7. The end concentration of methylene bridges increased as the molar ratio between formaldehyde and phenol increased. The concentration of methylene bridges is the lowest in synthesis 3 with the molar ratio of 1.8/1 because the temperature and pH value of the reaction mixture were too low.



**Figure 6.** Concentration profiles of hydroxymethylphenols in the synthesis of PF resins: synthesis 3 (molar ratio 1 : 1.8, pH=7); synthesis 4 (molar ratio 1 : 1.8, pH=8); synthesis 6 (molar ratio 1 : 2).



**Figure 7.** Concentration profiles of methylene bridges in the synthesis of PF resins: synthesis 6 (molar ratio 2 : 1); synthesis 2 (molar ratio 1.8 : 1); synthesis 3 (molar ratio 1.8 : 1, pH=7); synthesis 1 (molar ratio 1.6:1).

## Conclusions

Different PF resins were synthesized at different molar ratios. The characterization of the chemical structure of resole prepolymers and their intermediates is very complex because of the many closely related isomers available. Firstly, hydroxymethylphenols are formed by the initial substitution reactions between formaldehyde and phenol. Secondly, condensation reactions take place between hydroxymethylphenols themselves, as well as between hydroxymethylphenols and phenol. Methylene and methylene-ether bridges are formed in this step. These prepolymers contain many active hydroxymethyl groups. It was observed that the structure of the PF resin depends on the initial molar ratio of F/P, temperature and pH value.

In-line ATR-FTIR spectroscopy was found to be a successful and informative analytical tool for determining individual phenol and formaldehyde conversions as well as intermediate and final product composition. Absorbance bands which correspond to the PF resins are 1633, 1478 and 1116  $\text{cm}^{-1}$  for the C=C aromatic ring, C-H methylene bridge and C-O-C methylene-ether bridge, respectively.

## Acknowledgments

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## Povzetek

Sintetizirali smo različne fenol-formaldehidne prepolimere s spreminjanjem razmerja med formaldehidom in fenolom. Sestava fenolne smole je odvisna od začetnega razmerja monomerov, vrste katalizatorja, reakcijskih pogojev in koncentracije prostih monomerov na koncu reakcije. Pomemben vpliv na značilnosti sintetizirane smole imata tudi temperatura in pH vrednost pod katerimi poteka reakcija fenola s formaldehidom. Upoštevati moramo tri zaporedne reakcije : adicijo formaldehida na fenol, rast verige ali tvorbo prepolimera in končno zamreževanje ali reakcijo utrjevanja. Glede na pH vrednost dobimo dve vrsti prepolimera, novolak v kislem območju pH vrednosti, medtem ko dobimo rezol pod alkalnimi reakcijskimi pogoji. Rezolno smolo sintetiziramo z molarnim prebitkom formaldehida ( $1 < F/P < 3$ ). Produkti so mono - ali poli - jedrski hidroksimetil fenoli, ki so stabilni pri sobni temperaturi, vendar se pri povišani temperaturi zamrežijo v tridimenzionalen, netopen in netaljiv polimer.

Potek sinteze fenol - formaldehidnega prepolimera smo spremljali s pomočjo "in-line" ATR-FTIR spektroskopske analitske tehnike (ReactIR 4000), ki je opremljena z optičnim vodnikom in diamantnim kompozitnim senzorjem. Ta tehnika se je izkazala kot idealna za določanje prostega fenola in formaldehida, kot tudi njunih konverzij, prav tako pa tudi sprememb v sestavi prepolimera v odvisnosti od reakcijskega časa kondenzacije. Kinetični podatki, ki smo jih dobili s pomočjo ReactIR 4000 "in-line" reakcijskega analiznega sistema se dobro ujemajo s podatki določenimi s tradicionalnimi titracijskimi metodami. ReactIR tehnologija nadomešča časovno potratno in nenatančno "off-line" metodologijo.