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Preparation of high viscosity thermoplastic phenol formaldehyde polymers for application in reactive extrusion

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This work deals with the synthesis of high molecular weight, thermoplastic phenol formaldehyde (PF) resins for the application in the synthesis of polypropylene–PF compatibilizers by reactive extrusion. Phenol formaldehyde of high molecular weight is required in reactive extrusion with most polypropylene grades to meet the viscosity ratio (λ) requirement for best mixing. Special lab-grades of PF were therefore developed. The polymers synthesized were highly linear and in the M_w range of 10–30 000 g mole^{-1}. © 1998 Elsevier Science Ltd. All rights reserved.

(Keywords: phenolic resins; thermoplastic; high molecular weight)

INTRODUCTION

The field of polymer blends and alloys is growing both industrially and academically. The combination of two or more commercially available polymers through alloying or blending represents an inexpensive route for tailoring of a polymer compound. A well-compatibilized system results in better than additive improvement in physical properties such as impact strength and tensile strength[1,2]. However, most polymers are immiscible or insoluble in each other owing to the very low entropy of mixing for macromolecules. One important way of overcoming this immiscibility, and of controlling the blend morphology is through the use of compatibilizers, often block or graft copolymers, that act as interfacial agents in the polymer blends[3–7]. The chemical structure of these copolymers are composed of one part miscible with one blend component (polymer 1), and the other part with the other (polymer 2). They are amphiphilic. Usually, the blocks of the compatibilizer are either structurally identical with the blend components or contain functional groups complementary to the functionality of the components. In an ideal system the compatibilizer is, therefore, found at the interface between polymer 1 and polymer 2.

The compatibility of polypropylene (PP) with most polymers is poor due to the lack of polar/functional groups, and blending of PP with other polymers will require addition of a suitable compatibilizer. To prepare a copolymer compatible with PP and engineering polymers like polybutylene terephthalate, PBT, and polyphenylene ether, PPE, a phenol-like repeating unit of the polar part of the graft copolymer is expected to work. Phenolic resins (*Figure 1*) have important similarities in molecular structure to engineering polymers like polyphenylene oxide (PPO) polyphenylene ether (PPE) and polycarbonate (PC) and generally considered soluble or partially miscible with these

polymers. Polyethylene terephthalate (PET) and polybutylene terephthalate (PBT) might also be considered (*Figure 2*).

Thermoplastic phenol formaldehyde (PF) resins are characterized by their capability of forming hydrogen bonds with polymers containing carbonyl or carbonate groups[8], or by formation of covalent bonding if the polymer contains complementary groups to hydroxyl. In addition, the phenyl ring structure of phenolic resins are capable of forming secondary bonding to specific engineering polymers, by $\pi-\pi$ overlap.

In the synthesis of PP-graft-PF copolymers by reactive extrusion only high molecular weight PF novolaks (thermoplastic) were considered, to meet the viscosity ratio (λ) requirement for best mixing[9]. Most commercial novolaks are low molecular weight grades. Synthesis of special lab-grades was therefore required.

In this paper, the synthesis of high molecular weight, thermoplastic novolak polymers is demonstrated. In particular, the effects of various synthesis parameters have been studied. Statistical response surface techniques were used for experimental design generation, and for the analysis of main effects and interactions. One example of further application as compatibilizer is also demonstrated.

THEORY

Chemistry

Phenolic resins are polymers produced by condensation polymerization of phenol and formaldehyde. This group of polymers is commercially used in a broad range of applications. Phenolic resins can be divided into two main classes due to reaction variables such as pH of the catalyst, and the formaldehyde-to-phenol ratio (*Scheme 1*). Formaldehyde has a functionality of two. Phenol has a functionality of three; it will react at the position *para* and the two *ortho* to the hydroxyl. The two *meta* positions are generally considered unreactive. If a catalysed mixture of phenol and

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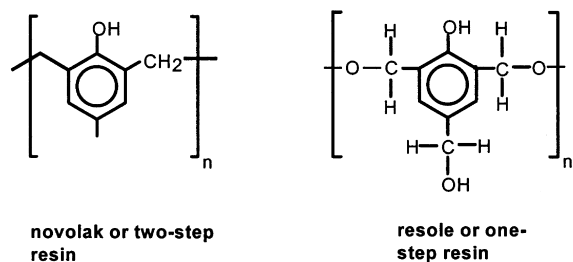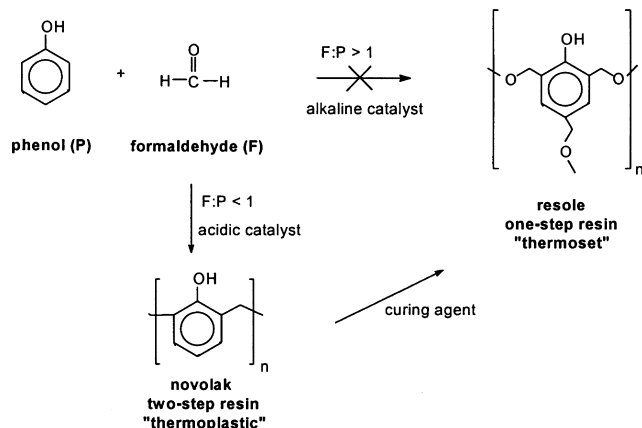
Figure 1 Structure of phenolic resin repeating unit

Scheme 1 Synthetic routes for *resoles versus novolaks*

formaldehyde contains one or more than one mole of formaldehyde per mole of phenol, it may react to produce a thermosetting resin. Resoles (one-step resin) are produced using an alkaline catalyst with a molar ratio larger than 1. If the reaction mixture contains less than one mole of formaldehyde per mole of phenol, it has sufficient functionality but not enough crosslinking molecules to be thermosetting and a thermoplastic resin results. The thermoplastic novolak resins are synthesized with a molar ratio less than 1 and with acid catalyst. To form highly linear phenolics, a bifunctional phenol such as *o*- or *p*-cresol, and/ or an *ortho*-directing catalyst (like divalent metal acetates) may be used.

Most commercially available thermoplastic PF polymers have weight average molecular weights (M_w) in the range of 500 to over 1000 g mole^{-1}. The molecular weight of phenolic resins depends on the formaldehyde-to-phenol ratio, the type of catalyst (acidic or alkaline), and the time and temperature of reaction. The most important factor for controlling the molecular weight of PF resins is the phenol-to-formaldehyde molar ratio. Commercial practice includes a molar ratio of formaldehyde to phenol within the range of (0.75–0.90):1. With weaker catalysts, it is possible to go slightly higher to 0.93 in molar ratio, but this is not very common. Even as high as 0.935 is reported by Baekeland[10], but only on a laboratory scale.
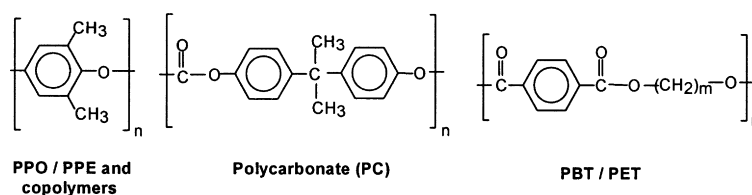
Application in reactive extrusion

In this present study, specific phenolic resins are synthesized for application in reactive extrusion with functionalized polyolefin material. Examples of functional groups are carboxylic acid anhydrides, carboxylic acids, epoxides, etc.

Phenol formaldehyde used as an ingredient in reactive extrusion needs to be thermoplastic to avoid uncontrolled crosslinking in the extruder. Therefore, only thermo-plastic PF polymers or so-called novolak resins were considered.

Only PF polymers with higher molecular weights were relevant for reactive extrusion with functionalized poly-olefin materials, to meet the required viscosity ratio for best possible dispersive mixing. The viscosity ratio (λ) should be near one at average shear rates for reactive extrusion, typically 100–1000 s^{-1}.

A possible way to achieve high molecular weight PF polymers is to increase the formaldehyde-to-phenol ratio towards unity. Other ways would be to use substituted phenol(s) alone or in combination with phenol.

EXPERIMENTAL

Procedure for preparation of novolak resins

Novolak resins were synthesized utilizing acid catalysed condensation techniques in water. A 37% aqueous solution of formaldehyde of the same quality was used in all trials. Multiple- component novolak resins were made by using solution condensation of various mixtures of phenol and substituted phenols such as resorcinol, *m*-cresol, *p-tert*-butyl phenol, 2-*tert*-butyl phenol, *o*- cresol, and formaldehyde. Oxalic acid was used as catalyst. In a typical reaction where formaldehyde was fed continuously (KLB.001), 470.6 g phenol (5.00 mole), 540.7 g *m*-cresol (5.00 mole), and 8.78 g oxalic acid (0.098 mole) were introduced to a five-necked polymerization flask (2000 ml, see *Figure 3*) and mixed to dissolve and carefully heated under nitrogen. During heating, keeping the temperature below 75°C, 729.7 g formaldehyde (9.00 mole) was carefully metered into the phenol/*m*-cresol-acid composition over 60 min.

When all the formaldehyde was added, the mixture was heated and refluxed (99–100°C) for 2 h. Then the reaction mixture was cooled to 82–85°C, and more oxalic acid (8.78 g, 0.098 mole) was added. The mixture was heated to reflux (99–100°C) for 25 h, and then the volatile components were distilled off. During the distillation, the temperature was gradually increased and the pressure was gradually decreased until final conditions of 215°C/10–15 mmHg was achieved. Throughout the reaction, the reaction vessel was equipped with a double surface condenser. The novolak was then flaked and ground to appropriate size.

Similar procedures were used for all resins with continuous feeding of formaldehyde (C in *Table 1*).

For resins with discontinuous formaldehyde feeding (DC

Figure 2 Structural units of important engineering polymers with structural similarities to PF repeating unit
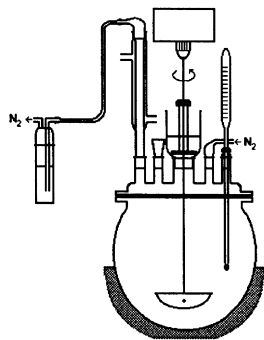
Figure 3 Reaction vessel for the synthesis of PF novolaks

statistical experimental designing three main types of variables exist: quantitative, qualitative and mixture variables. Quantitative variables are continuous in a given range, such as temperature or concentration. Qualitative variables have discrete values, such as manufacturer or additive type. Lastly, mixture variables are quantitative (or continuous) variables which assume values between 0 and 1, and which sum to unity. In this present design the various phenol derivatives were all mixture variables; they were quantitative variables with values between 0 (0 mole%) and 1 (100 mole%).

Formaldehyde feeding was a qualitative variable with values of C (continuous) or DC (discontinuous), and formaldehyde to phenol molar ratio a quantitative variable

Table 1 Novolak synthesis design

Resin code	Mole ratio CH_2O^a	CH_2O feeding[b]	Mole fraction of phenol and phenol derivatives in feed[c]					
			Resorcinol	*m*-cresol	*p-tert*-butyl-phenol	2-*tert*-butyl-phenol	*o*-cresol	Phenol
KLB.001	0.90	C	0.00	0.50	0.00	0.00	0.00	0.50
KLB.002	0.94	C	0.02	0.00	0.08	0.00	0.00	0.90
KLB.003	0.90	DC	0.00	0.50	0.00	0.00	0.00	0.50
KLB.004	0.90	DC	0.01	0.215	0.047	0.068	0.089	0.571
KLB.005	0.94	DC	0.02	0.00	0.08	0.00	0.00	0.90
KLB.006	0.90	DC	0.00	0.00	0.10	0.00	0.00	0.90
KLB.007	0.90	C	0.00	0.00	0.10	0.00	0.00	0.90
KLB.008	0.90	DC	0.02	0.00	0.00	0.15	0.20	0.63
KLB.009	0.94	DC	0.00	0.00	0.00	0.00	0.20	0.80
KLB.010	0.94	C	0.00	0.00	0.00	0.00	0.20	0.80
KLB.011	0.94	DC	0.02	0.50	0.00	0.00	0.00	0.48
KLB.012	0.90	C	0.02	0.00	0.00	0.15	0.20	0.63
KLB.013	0.94	C	0.00	0.00	0.00	0.15	0.00	0.85
KLB.014	0.94	DC	0.00	0.00	0.00	0.15	0.00	0.85
KLB.015	0.90	C	0.02	0.50	0.10	0.15	0.20	0.03
KLB.016	0.90	DC	0.02	0.50	0.10	0.15	0.20	0.03
KLB.017	0.94	C	0.02	0.50	0.00	0.00	0.00	0.048
KLB.018	0.94	DC	0.02	0.50	0.10	0.15	0.20	0.03
KLB.019	0.94	C	0.02	0.50	0.10	0.15	0.20	0.03

[a]Mole ratio CH_2O to phenol and phenol derivatives (Σ mole phenol and phenol derivatives = 1.00)
[b]C means continuous feeding, DC means discontinuous feeding of CH_2O
[c]Total amount sum to unity

in *Table 1*), all reactants were mixed in the preheating stage, except for the second feed of oxalic acid. In a typical reaction where formaldehyde was fed discontinuously (KLB.003), 470.6 g phenol (5.00 mole), 540.7 g *m*-cresol (5.00 mole), 729.7 g formaldehyde (9.00 mole), and 8.78 g oxalic acid (0.098 mole) were introduced to a five-necked polymerization flask (2000 ml, see *Figure 3*) and mixed to dissolve and carefully heated under nitrogen. The mixture was heated and refluxed (99–100°C) for 2 h. Then the reaction mixture was cooled to 82–85°C, and more oxalic acid (8.78 g, 0.098 mole) was added. The mixture was heated to reflux (99–100°C) for 25 h, and then the volatile components were distilled off. A similar procedure to the continuous conditions was followed for the remaining steps.
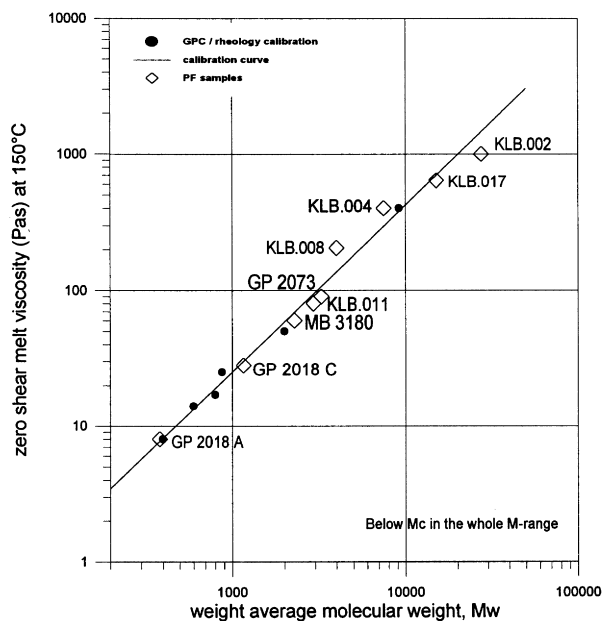
Experimental design

The synthesis was conducted according to an experimental design set-up, using response surface modelling. Design generation and analysis was done on ECHIP 6.08 software, Windows version. *Table 1* presents the experimental design with various specifications of each design trial. In

ranging from 0.90 to 0.94. Partial least square (PLS) regression analysis was done using Baccos v 3.0 software developed at Borealis. This is a program for background correction of spectral data, and offer a strong latent variable calibration technique.

Procedure for preparation of polypropylene–phenol formaldehyde graft copolymers

Polypropylene–phenol formaldehyde graft copolymers were prepared by a continuous reactive extrusion process. Three maleic anhydride (MAH) functionalized materials, PB 3002, PB 3150 and E-43, with various MAH content and viscosity provided the polymers. All grades are commercially available from Uniroyal Chemical (PB-grade) or Eastman Chemical Company (E-grade). PP-g-GMA is a glycidyl methacrylate functionalized PP, an in-house experimental grade offering a GMA content of 0.8 wt%[11–13].

The functionalized PPs and PF polymer coded KLB.002 were vacuum-dried at 100°C for 12 h before compounding to remove residual monomers and traces of water.

Figure 4 Zero shear melt viscosity (150°C) plotted *versus* weight average molecular weight for experimental and commercial novolak grades

Table 2 Viscosity, weight and number average molecular weight for different resin codes

Resin code	η_0 150°C/(Pa)	M_w^a(g mole^{-1})	M_n^a(g mole^{-1})
KLB.001	45	1400	630
KLB.002	1000	27550	3520
KLB.003	35	1600	730
KLB.004	80	2950	875
KLB.005	710	19100	2105
KLB.006	20	850	307
KLB.007	32	1160	475
KLB.008	37	1265	535
KLB.009	205	4000	1270
KLB.010	450	9100	1923
KLB.011	435	7700	1520
KLB.012	54	2100	930
KLB.013	275	5400	1375
KLB.014	205	4000	1150
KLB.015	15	600	225
KLB.016	12	530	210
KLB.017	640	15100	2830
KLB.018	310	6300	890
KLB.019	480	8100	1730

The preparation of the PP–PF graft copolymers were conducted in a 25 mm Clextral BC 21 intermeshing corotating twin screw extruder (TSE) with 12 barrel segments and an overall length, L/D of 44. Functionalized PP was fed into barrel 1 and PF resin into barrel 3, at a total throughput of 3 kg h^{-1}. The screw rotation speed was 200 rpm and the barrel set temperature 190–195°C. Average residence (reaction) time at these conditions was 2.5 min. The extruder was equipped with highly efficient vacuum venting to remove unreacted species and reaction by-products. Inert atmosphere (nitrogen) was used to reduce polymer degradation. The extrudate was immediately quenched in a water bath and then pelletized.

The extruder screw profile was configured to allow a good melting of the polymers followed by efficient mixing and high shear, high residence time in the reaction zone, and venting at the outlet of the extruder. The extruder was equipped with a K'tron loss-in-weight feeding system for accurate feeding of raw materials.

Characterization of the novolak resins

Molecular weights of the PF resins were determined using rheology and gel permeation chromatography (g.p.c.).

Samples were analysed on a Bohlin CS melt rheometer and a Waters 150 CV GPC instrument. Molecular composition of the PF polymer was also analysed by ^{13}C NMR using a Bruker 500 MHz ^{1}H NMR-instrument, and at solid state mode.

Characterization of the polypropylene–phenol formaldehyde graft copolymers

A systematic characterization of the reaction products of the functionalized PP with the PF was undertaken by thorough selective extraction followed by sample preparation and spectroscopic analysis. Infrared analysis was performed on a Perkin Elmer 1725 × FTIR instrument using transillumination.

The extraction method and solvent were adapted to this specific polymer system, and according to solubility test of neat polymers, acetone was a chosen as a selective solvent for PF polymers. Sufficient extraction time was 48 h on pellets followed by 48 h on film. The film was then repressed for *FT*i.r. analysis. Both extracted and non-extracted samples were analysed. Materials (film) were predried before *FT*i.r. analysis at 100°C for 2 h. Films used in this study were sufficiently thin to be within an absorbance range where the Beer–Lambert law is obeyed (<0.6 absorbance units). Polymer films were made using a Pasadena hydraulic press and a distance frame of 0.1 mm.
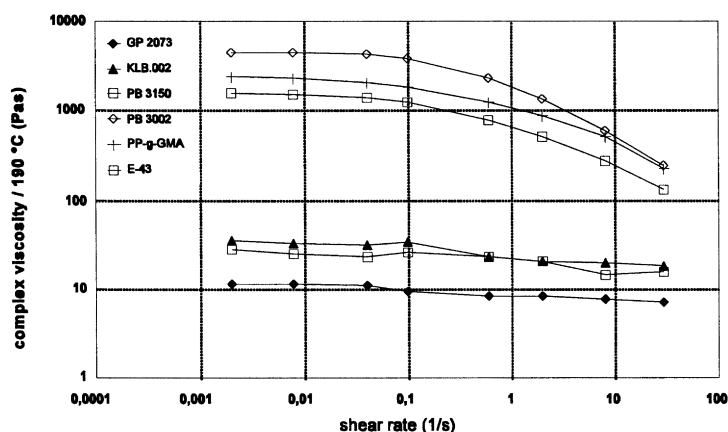
Figure 5 Complex melt viscosity (190°C) plotted *versus* shear rate for experimental and commercial novolak and PP grades
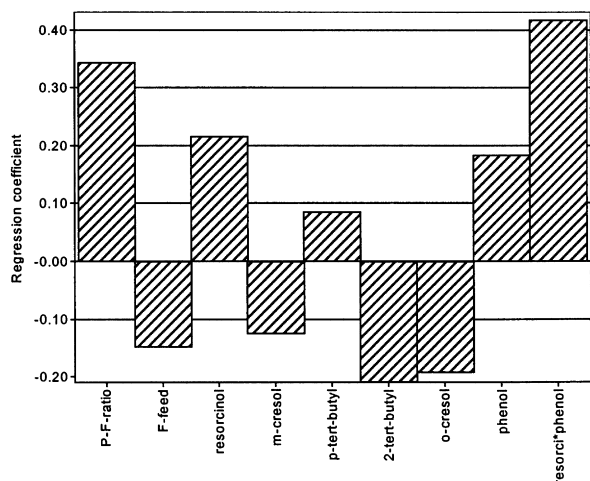
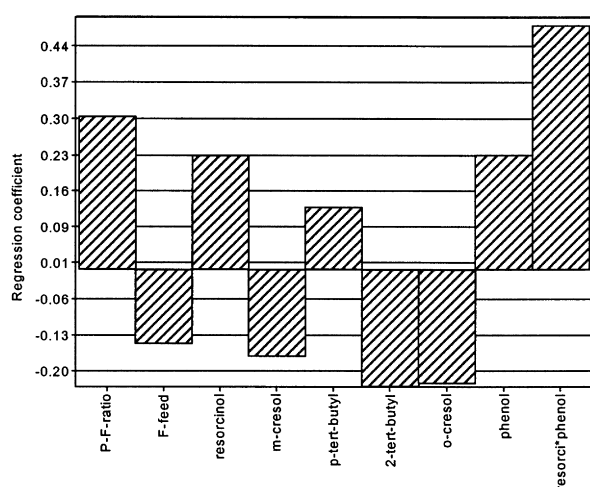**Figure 6** Regression coefficients, $\eta_{0.05}$ is the response

Figure 7 Regression coefficients, M_w is the response

For quantitative analysis of the amount of reacted PF polymer, data were obtained from the *FT*i.r. spectrum of extracted samples, using the peak height ratio of the C=C peak (ring stretch) at 1610 cm^{-1} to that for the C–H peak (bend) at 973 cm, and controlled by a corresponding peak height ratio of C=C peak (ring stretch) at 1641 cm^{-1} to that for the C–H peak (bend) at 899 cm^{-1}. These ratios were used as a measure for the relative content of PF in the graft copolymer. Since the PF is rather a high molecular weight species, the content of PF in the samples prior to extraction was assumed to be equal to the feeding content.

RESULTS AND DISCUSSION

Novolak resins with different average weight molecular weights (M_w) were synthesized, using solution condensation of resorcinol, *m*-cresol, *p-tert*-butyl-phenol, 2-*tert*-butyl-phenol, *o*-resol and phenol with continuous or discontinuous feeding, using an acid catalyst.

The zero shear melt viscosity at 150°C, weight and number average molecular weight are listed in *Table 2*. The zero shear melt viscosity of the experimental grades is compared with commercially available, medium-to-high molecular weight novolaks (coded with prefix GP and MB) in *Figure 4*.
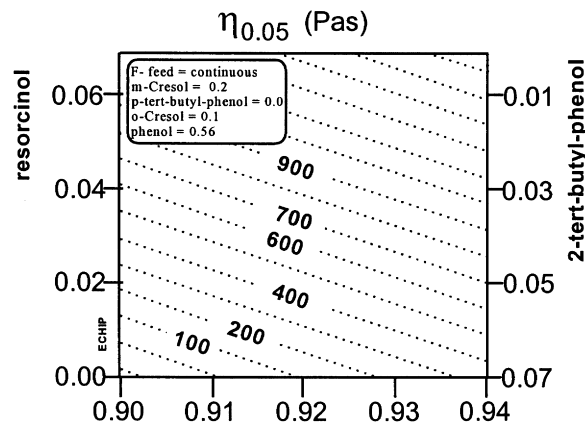

Figure 8 2-D response surface plot of $\eta_{0.05}$ at fixed synthesis component settings and continuous feeding of formaldehyde
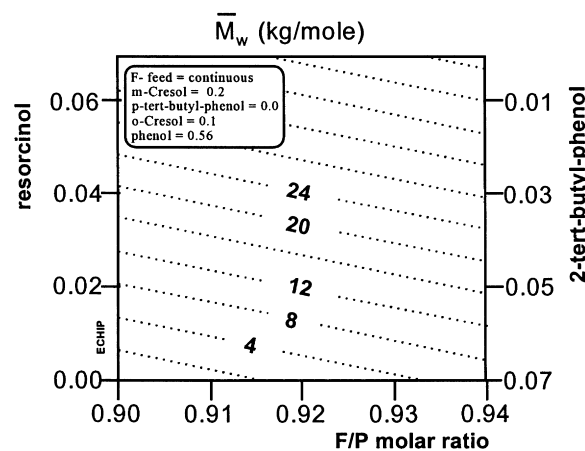

Figure 9 2-D response surface plot of M_w at fixed synthesis component settings and continuous feeding of formaldehyde

In *Figure 5* are shown the dynamic melt viscosity at 190°C of various functionalized PP grades, commercial high molecular weight PF, and experimental grade novolaks. The grades PB 3002, PB 3150 and E-43 are all MAH functionalized PP with increasing maleic anhydride content, and decreasing viscosity. PP-g-GMA is a glycidyl methacrylate functionalized PP.

Figures 6 and 7 are plots of the PLS regression coefficients of the responses $\eta_{0.05}$ and M_w, respectively. The figures indicate that the F–P ratio term, and the crossterm of resorcinol*phenol were the most important synthesis parameters for $\eta_{0.05}$ and M_w. Resorcinol and 2-*tert*-butyl phenol also seemed to influence the molecular weight significantly. The first three variables were positively correlated to $\eta_{0.05}$ and M_w, while 2-*tert*-butyl phenol was negatively correlated.

A 2-D response surface plot of $\eta_{0.05}$ and its variation with F/P molar ratio, and mole ratios of resorcinol and 2-*tert*-butyl phenol is shown in *Figure 8*. Feeding was done continuously, and all other variables were fixed at the levels indicated in the figure. No global or local maximum or minimum conditions were predicted within the examined process window. However, within the experimental frame the highest $\eta_{0.05}$ (1750 Pa) was obtained at a F/P of 0.94, a resorcinol ratio of 0.08 and 2-*tert*-butyl of 0. *Figure 9* indicates that the highest M_w (50 kg mole^{-1}) was predicted in the same conditions. These mixture design plots are different from well-known triangle mixture plots, owing to
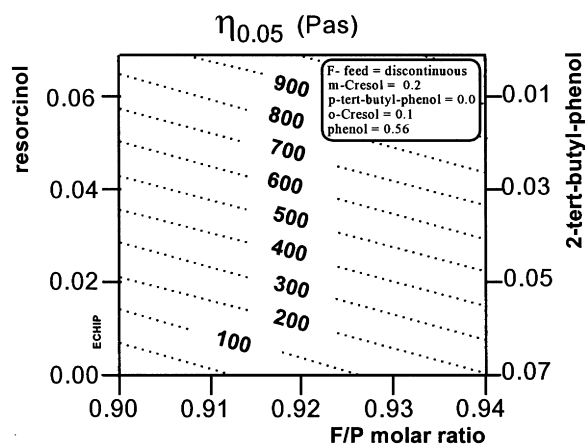
Figure 10 2-D response surface plot of $\eta_{0.05}$ at fixed synthesis component settings and discontinuous feeding of formaldehyde
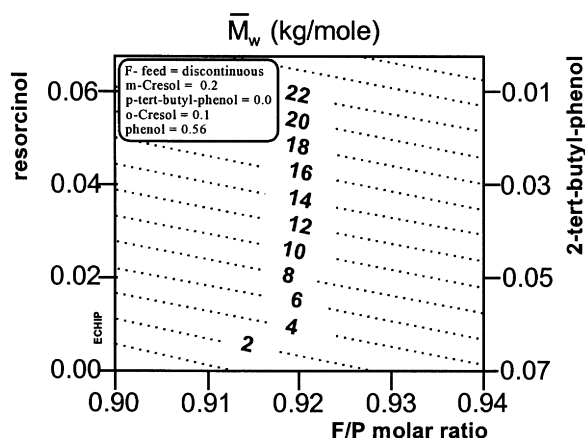

Figure 11 2-D response surface plot of M_w at fixed synthesis component settings and discontinuous feeding of formaldehyde

Similar plots are shown for discontinuous feeding in *Figures 10 and 11* for $\eta_{0.05}$ and M_n, respectively. Again the highest predicted $\eta_{0.05}$ and M_w were obtained at the same variable settings as for the case of continuous feeding. However, the level was generally lower for this feeding technique giving $\eta_{0.05}$ of 1300 Pa and M_w of 32 kg mole^{-1}.

From the g.p.c. analysis very low concentrations of crosslinking or severe branching of the PF resins were detected for all of the analysed resins. The structure was mainly linear.

Solid state ^{13}C n.m.r. analysis was also conducted to check the proposed linear structure. A solid state ^{13}C n.m.r. spectrum of the KLB.002 grade is shown in *Figure 12*. The spectrum indicates rather broad lines due to the general difficulty of running solid state n.m.r. of such complex polymers. However, from the spectrum it is evident that the polymer contains four main substituted C atoms. The analysed KLB.002 resin consisted of 90% (mole) of phenol, 2% (mole) resorcinol and 8% (mole) *p-tert*-butyl phenol. The hydroxyl-substituted carbon atoms (phenolic-OH) absorbed at about 150 ppm, as indicated in the spectrum. Further upfield, the carbon atoms in the *m*- and *p*-positions absorbed at about 128 ppm. The carbon atoms in the *o*-position and in the methylene bridge were detected at about 114 and 33 ppm, respectively. Three non-substituted positions on the benzene ring were detected and, from this observation, it was evident that the PF molecules of KLB.002 were highly linear in structure.

Possible reaction routes of PP polymer with maleic anhydride, and epoxy functionalized PP are shown in *Figure 13*. Two possible mechanisms are indicated for epoxy functionalized PP, depending on the presence of base-catalyst (ii), or not (i).

A comparison of reactivity of the PF grade coded KLB.002 with three different MAH and one GMA functionalized PP grades is given in *Figure 14*[14,15]. The
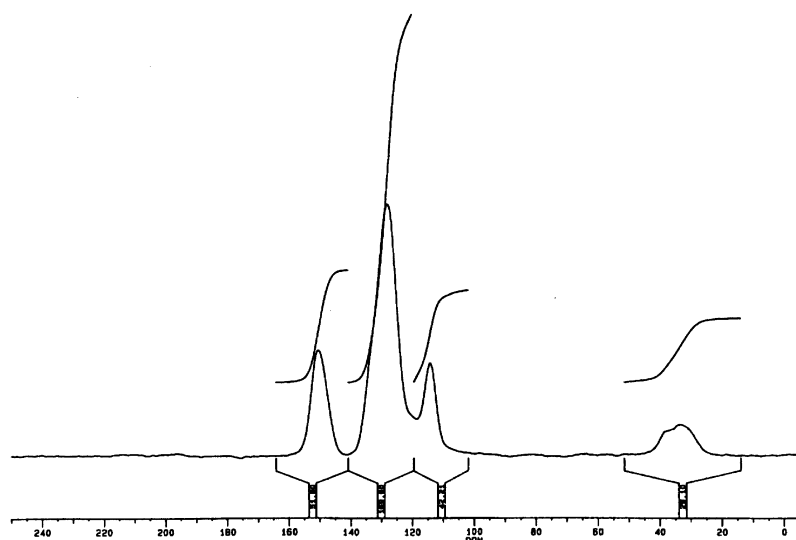

Figure 12 ^{13}C NMR of KLB.002

the combination of mixture and non-mixture variables in this present design. As may be seen, the plot is instead a rectangular plot with two complementary vertical axes whose scales run in opposite directions. The mixture coordinates on the plot sum to $1 - \sum$ [*non-axis mixture variables*].

procedure for synthesis and characterization is described in the experimental part of this paper. Very low contents of unreacted PF resins were detected in the products, as determined by selective extraction methods. *Figure 14* clearly shows that the examined PP-g-GMA had higher reactivity and/or more available groups than the PP-g-MAH
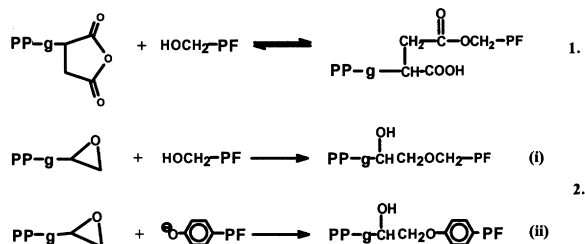
Figure 13 Reaction of functionalized PP and PF
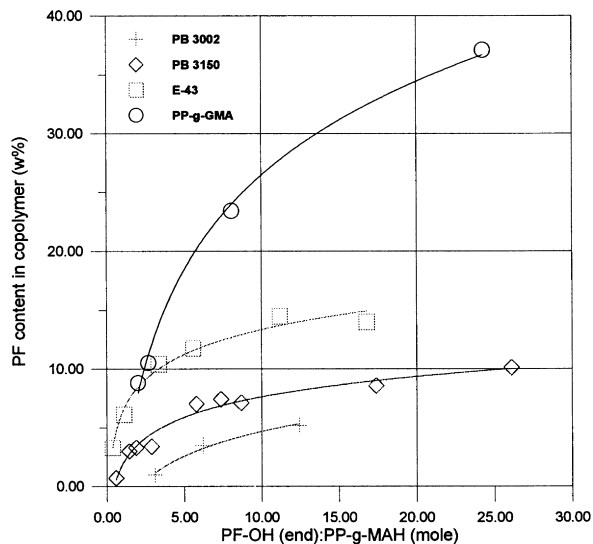

Figure 14 Variations in PF content of the PP-PF graft copolymer for various OH:MAH ratios. PF grade used is KLB.002

grades. The high functionality, low viscosity PP-g-MAH grade E-43 resulted in the highest PF content of all of the evaluated PP-g-MAH grades.

CONCLUSION

High molecular weight novolaks for application in reactive extrusion were synthesized with M_w ranging from 530 to 27550 g mole^{-1}. Continuous feeding, higher F/P molar ratio, and resorcinol were the dominating synthesis variables for obtaining PF resins of high molecular weight.

Despite the high molecular weights of the synthesized PF resins, they were all analysed to have a linear structure by g.p.c. and ^{13}C n.m.r.

In reactive extrusion experiments with various functionalized PPs, these high molecular weight PF and standard low molecular weight grade PF were compared. In these experiments high molecular weight novolaks were found to be very effective reactants for making PP–PF graft copolymers.

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