Urea formaldehyde resin pdf

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Ding, Z., Ding, Z., MA, T. and Zhang, H. (2020). "Condensation reaction and crystallization of urea-formaldehyde resin during the polymerization process", biores. 15 (2), 2924-2936.urea-formaldehyde (UF) Resins have been summarized with different molar ratios and solid content, and at the same time have been treated in conditions of different pH values. The cured UF resin polymerization behaviors were examined by synchronous thermal analysis (TG-DSC). The crystallinity of the cured UF resin was analyzed by X-ray diffractometry. The cared UF resin gel time was recorded with chemical methods. The results indicate that the condensation reactivity carries the condensation reaction and crystallization to reproduce various roles during the UF resin polymerization process. The reaction and crystallization of condensation in the polymerization process interact to cause different structures of cured resin. There is therefore a new mechanism for the polymerization of the UF resin (reaction crystallization). Download PDFFULL ArticleComondensation Reaction and urea-formaldehyde resin crystallization During the Zhongjian Ding polymerization process, A, * Zhongqiang Ding, B, * Tianlin MA, AÃ ¢ and Hua Zhang A Urea-formaldehyde resin (UF) has been synthesized with Several molar and solid relations contained, and simultaneously were treated in conditions of different pH values. The cured UF resin polymerization behaviors were examined by synchronous thermal analysis (TG-DSC). The crystallinity of the cured UF resin was analyzed by X-ray diffractometry. The cared UF resin gel time was recorded with chemical methods. The results indicate that the condensation reactivity carries the condensation reaction and crystallization to reproduce various roles during the UF resin polymerization process. The reaction and crystallization of condensation in the polymerization process interact to cause different structures of cured resin. There is therefore a new mechanism for the polymerization of the UF resin (reaction crystallization). Keywords: urea-formaldehyde resin; Polymerization; Condensation reactivity; Condensation reaction; Crystallization; Crystallinity contact information: A: College of Materials Science and Chemical Engineering, University of Chuzhou, 239000, Anhui Province, China; B: Singapore Institute of Technology, Singapore; * Corresponding authors: zjing@chzu.edu.cn; ding.zhongqiang@singaporetech.edu.sg introduction The urea-formaldehyde resin (UF resin) is one of the most important adhesives for the assembly of the wooden-based panels made by man, such as MDF, plywood and particle. Commonly, the resin of the thermosetting-formaldehyde (UF) is curated by the condensation reaction in acid conditions. The UF resin polymerization process has been studied using the calorimetry technology of differential scan (DSC)). SzesztayÃ ¢ et al .Ã ¢ (1993, 1996) found that an exothermic peak (condensation reaction) and an endothermic peak (decomposition of methylene-ether connections) appeared in the DSC curve. In a high-pressure DSC cell, the endothermic effect moved to higher temperature and avoided to disturb the exothermic peak. XingÃ ¢ et alÃ ¢ (2005) has studied the effect of the catalyst content on the UF resin polymerization behavior in high pressure conditions. The parameters of the exothermic peak have shown that the reactivity of the resin condensation has increased by increasing the pH value in the resin system. The Pizzi team (2019) analyzed the effect of the initial molar ratio on the reaction heat, debut temperature, exothermic and endothermic peak temperatures of the DSC curve. Results showed that the initial molar ration obviously influenced the physical-mechanical proprieties of the cards. Studies also discovered that cured UF resin has a crystalline state that does not exist in other thermosetting resins such as phenolic resins, epoxy resins, etc. (GuptaÃ ¢ et al. 1985). The crystal clear structure of the UF resin is concerned with the UF resin, especially in the UF resin with low formaldehyde molar reports to the urea (molar ratio f / u) (NuryAwanÃ ¢ et al.2016). The crystallinity of the cured resin is influenced by molar reports, from the temperature of process and curing agents. The cured UF resin crystallinity increases as the molar ratio of f / u decreases (park and Causin 2013; SINGHA ¢ et al. 2014; NURYAWANÃ ¢ et al. 2017). The crystallinity of the cured resin was inversely proportional to the degree of branching of the cured resin structure (NuryawanÃ ¢ et al. 2015; Park and Jeong 2011; Ding and Tian 2017). On the basis of previous studies, there is not only a condensation reaction but also the crystallization during the polymerization process of the UF resin. The impact of factors such as the molar ratio of f / u and the polymerization agent, etc., On reactivity or crystallinity of the condensate was reported. However, the study of the combined effects of the condensation reaction and the crystallization on the UF resin curing mechanism is very limited. Therefore, in the present work, the UF resins with a different reactivity of the condensation has been obtained by adjusting the molar ratio F / U F / U, the solid content and the pH value of the polymerization condition. The time of the gel, which characterizes the responsiveness, as well as the crystallinity of the cured resin, which characterizes the structure of the resin, were measured under the same pH conditions. The TG-DSC curves were obtained under the normal pressure. They were studied the polymerization of UF resins with different reactivity of the condensation processes. The results clarify the variety of the UF resin polymerization mechanism with different reactivity of condensation. The accurate understanding of the UF resin curing mechanism is advantageous for the development of UF resin with low formaldehyde release and high performance. Experimental Materials formaldehyde reagent grade (37%), sodium hydroxide, formic acid and hydrochloric acid were purchased from Shanghai Reagent Chimical Ltd., China. Urea industrial grade has been provided by Chuzhoujinhe Chimical Industry Ltd., China. Preparation of UF resins All resins were synthesized in the laboratory. The synthesis of all of the UF resin was a typical three-step procedure (alkaline-acidic-alkaline). First, the formaldehyde (f = 250 g) has been poured into a flask with three points and adjusted to pH 7.5 to pH 8.0 with sodium hydroxide solution 20%. The first amount of urea (U1 = 94.4 G) has been added to give the molar ratio of f / u 2.0. The mixture has been heated to 90 Â ° C and kept at this temperature for 30 minutes. The condensation reaction took place by adding formic acid (10%) to obtain a pH from 4.6 to 4.8 until the endpoint was reached 1. The pH was adjusted to 5.0 with hydroxide solution sodium, and the second amount of urea (u2 = 23.5 g) has been added to impart the molar ratio of f / u of 1.60. The temperature was kept at 85-90 Â ° C until it has been reached the endpoint 2. The endpoint was determined looking cloudy solution when the mixture has been allowed to fall into a beaker of water under certain temperature (Endpoint 1: 20 Â ° C, Endpoint 2: 40 Â ° C). When Endpoint 2 has been reached, the pH was adjusted to 7.5 to 8.0 with the sodium hydroxide solution. It was adding a third amount of urea (U3). The reaction mixture was kept at 70 Â ° C for 30 minutes. Finally, the mixture has been cooled to room temperature. The pH was adjusted to 7.5 to 8.0 again. Various amounts of U3Ã ¢ (U3 = 0 G, 16.9 G, 39.4 G, 61.9 G, or G 80.8) have had different molar ratios F / UF / U (the molar ratio F / U = 1.60, 1.40, 1.20 1.05 and 0.95, respectively). The UF resin with the molar ratio F / U of 1.60 has been dried on the vacuum conditions to obtain resins with different solids contents (50%, 58% and 67%). The solid content of the UF resin was measured according to GB / T14732 (2006). Preparation of samples for The liquid resin with different pH values The pH values were adjusted to 7.5, 4.5, 4.0 and 3.0 with sulfuric acid solution (20%). In other studies, the PH value of the UF resin used to be adjusted by ammonium salt, such as NH4CL. The pH value was influenced by the formaldehyde produced during the UF resin polymerization process. This meant that the pH value has changed with the variety of the quantity of formaldehyde during the polymerization process (XingÃ ¢ et al. 2005; dazmiriÃ ¢ et al. 2019). 2019). Keep the pH values during the UF resin polymerization process, the sulfuric acid solution was used to directly adjust the pH values. Powder powdered resin Resins with different pH values were treated in the oven at 120 Â ° C for 2 hours. The cured resins were ground in particles and sifted to obtain particles between 80 mesh and 120 mesh for the XRD test. Gel time measurement The liquid resin gel time with different pH values was measured at 100 Â ° C from a gel time meter (Sunshine 22a, Shanghai, China). An average of three replicas has been used for each measurement. TG-DSC measurement TG-DSC measurements were performed with a synchronous thermal analyzer (SDT Q600, TA, City, USA) with the 5K / min heating rate in dynamic nitrogen atmosphere (100 ml / min). The mass of the samples was from 10 to 15 mg. The temperature of the samples varied from 30 ° C to 200 Â ° C. The TG-DSC test was carried out with an open crucible. All the samples were the same as those in the test of the gel time. X-ray diffraction (XRD) A X-ray diffractometer (D8 Advance, Bruker, Karlsruhe, Germany) was used to investigate the crystallinity of the UF resins taken care of. The cured powder resins were analyzed at room temperature using a source of X-rays Cuka-1 with a wavelength (Åž â € ") of 1,5406 ãƒ .... the angle of incidence has changed from 10 Â ° at 70 ° in steps of 0.02 Â ° / min. The XRD DiffractGram was analyzed by the MDI Jade5.0 software. The crystallinity of the refined UF resin has been obtained. Results and discussion TG-DSC curves of the resin of the urea-formaldehyde The UF resin was cured with the molar ratio f / u of 1.60 and solid content by 50% in conditions of pH 7.5 and 3.0, respectively. Figure 1 shows the TG-DSC curves of the UF resin polymerization. There are two endothermic peaks (peak 1 and peak 2) on each DSC curve, and these peaks are accompanied by significant mass losses that appear on the TG curve. Under the pH 7.5 condition, the water in the initial resin is quickly evaporated with temperature increment before peak 1. Due to the loss of the water, the liquid state of the resin changes to the state of the gel between the peak 1 and the Peak 2 (NuryAwanÃ ¢ et al. 2017). The water resistance that spreads from interiors to the resin surface in gel state increases and water evaporation rate decreases. When the temperature reaches the peak 2, the formaldehyde from the decomposition of methylene-ether connections, as well as the small amount of initial water, is evaporated by the resin, accompanying the endothermic effect (SzesztayÃ ¢ et al. 1993). Temperatures at peak 1 and endothermic heat at pH 3.0 are lower than those at pH 7.5. This indicates that the condensation reaction took place. The condensation reaction is an exothermal reaction. The exothermic heat compensates for an endothermic heat. No exothermic peak appears between peaks 1 and 2 on the DSC curve because the exothermic effect is covered by endothermic effect of water evaporation (SiimerÃ ¢ et al. 2003; ZorbaÃ ¢ et al. 2008). The condensation reaction will increase the degree of cross-linking of the resin. A higher degree of cross-linking in the structure leads to greater resistance to diffusion. More water (reaction water) and methylene-ether connections are produced by condensation reaction. Therefore, it is known that there will be greater quantities of water and methyluene-ether connections in the resin. Here, comparing with the TG curves of pH 3.0 and 7.5 between peaks 1 and 2, the water evaporation rate at pH 3.0 is obviously lower than that at pH 7.5. The mass loss for peak 2 at pH 3.0 is more than the pH 7.5. This means that more than the initial water, reaction water and methylene-ether connections are left in the resin in the Gel before peak 2. However, these substances are all quickly evaporated at peak 2 (SiimerÃ ¢ et al. 2003; DazmiriÃ ¢ et al.. 2019). Figure. 1. The TG-DSC curves of the UF resin with a molar ratio f / u of 1.60 and solid contents of 50% of figure 2 shows the TG-DSC curves for when the UF resin with a pier ratio of 1.60 and various solid content (50%, 58% and 67%) were treated under the condition of pH 3.0. 3.0. 3 shows the gel time and the mass loss of resins corresponding to peak 2. The gel time has decreased with the increase in solid content. This means that the resin condensation reactivity has increased with the increase in solid content. Figure 2 also shows that the quantity of the substance evaporated at peak 2 (mass loss) has increased with the increase in condensate reactivity rather than increasing the water content of the initial resin. This indicates that the evaporated substance at peak 2 was mainly derived from the reaction water, as well as due to formaldehyde from the decomposition of methylene-ether connections. Fig. 2. TG-DSC curves of the UF resin with the molar ratio f / u of 1.60 and contents of different solids under pH 3.0 conditions fig. 3. It is time of gel and mass loss corresponding to the peak 2 of the resin UF with molar f / u The ratio between 1.60 cratarization In condition of pH 3.0 Figure 4 shows that the temperature at peak 2 (TP) increased with the increase in the resin gel time under pH 3.0. This means that TP has decreased with the increase in resin condensation reactivity. The studies found that, for the UF resin with higher f / u molar ratios, the increase in condensation reactivity could lead to a greater degree of branching of the resin. The branched network structure has facilitated the penetration of water in the interior of the cured resin, which led to more hydrolysis (park and Jeong 2011; Ding and Tian 2017). In other words, the branched network structure facilitates water and formaldehyde to evaporate from interiors of the resin in the gel state at lower temperature as well (SiimerÃ ¢ et al. 2003). The value of TPÃ ¢ TP is inversely proportional to the resistance to the diffusion of the evaporation of the substance in the resin. It is reasonable to conclude that TPÃ is influenced by condensation reactivity, from the rail process and the degree of branched structure of the polymerized resin. Figures. 4. It is the Time del Gel and the TPÃ, peak 2 of the UF resin with the Molar ratio f / u of 1.60 lending in conditions of condensation reaction of pH 3.0 and crystallization of the urea-formaldehyde resin during the process of Polymerization in conditions of different figure pH 5 shows the pH process. Peak 2 of the DSC curve, when the UF resin with the 1.60 molar ratio was hardened under conditions of pH 3.0, pH 4.0 and pH 4.5, respectively. TPÃ, TPÃ, peak 2 is decreased with the decrease of pH values. This means that water and formaldehyde in resin evaporated more easily with the increase in the reactivity of the condensation of the UF resin. However, figure 6 shows that the TP is increasing with the decrease of the pH value, when the UF resin with the molar ratio f / u of 0.95 was hardened in conditions of pH 3.0, pH 4.0 and pH 4.5 , respectively. This means that it is difficult to evaporate water and resin formaldehyde with the increase in the reactivity of the condensation of the UF resin. In summary, there are several impacts of the condensation of the UF resins condensation on the structure of the resin taken care of with different Molar ratios f / u. Figure 7 (a) shows that, for the UF resin with the Molar ratio f / u of 1.60, the structure of the UF resin cured was amorphous. Figure 8 shows that the crystallinity of the UM cured was close to zero and that has not been influenced by the pH value. Only the value of TPÃ has been influenced by the pH value. This indicates that the condensation reaction has improved the degree of ramification of the UF resin cured and limited the crystallization of the UF resin during the polymerization process. The condensation reaction played an important role during the polymerization process for the UF resin with the molar ratio f / u of 1.60. Figure. 5. Peak 2 on DSC of the UF resin with a Molar ratio f / u of 1.60 cribration with different pH fig. 6.Ã ¢ At peak 2 on the DSC curves of the UF resin with the molar ratio f / u of 0.95 cure the figures 7 (B) and 8 shows that, for the UF resin with the molar ratio f / u of 0.95, the crystallinity of the cured resin has increased with the decrease of the pH value. This means that the reactivity of the UF resin condensation has improved crystallinity crystallinità Decreased the degree of branched structure of hardened resin. As we said, the condensation reactivity of UF resin with a low molar ratio was lower than that with a high molar ratio (Parka et al. 2006). When UF resin with the molar ratio f / u of 0.95 healed, the macromolecular chains first intertwined with each other and are orderly arranged due to the lower reactivity. The crystalline structure has been formed in hardened resin (at the. Nuryawanã, ET 2017). The condensation reaction between the adjacent verified reactive groups in the crystal region. New covalent bonds fixed the molecular chains in the crystal area and so the crystallinity increased. The agglomeration and crystallization occurred before the condensation reaction. In other words, the crystallization played an important role during hardening for the UF resin with the molar ratio f / u of 0.95, but the condensation reaction only improved the crystallization. Fig. 7.a X-ray diffractograms of UF resins hardened with f / u Molar ratio of 1.60 and 0.95 in different pH conditions fig. 8.a TPA OFA peak 2 and crystallinity of resin hardened with f / u Molar ratio of 1.60 and 0.95 in different conditions of pH condensation of reaction and crystallization of urea-formaldehyde resin with different molar ratio during process vulcanization Figure 9 shows the peak 2 of DSC curves, when UF resins with different molar ratios (1 , 60, 1.40, 1.20 and 0.95, respectively) were treated with the condition of pH 3.0. The OFA TPA value increases when the molar ratio f / u decreased. This means that the evaporation resistance of water and resin formaldehyde increased with the decrease of f / u molar reports. Figure 10 shows that the crystallinity of the hardened UF resins obviously increased with the decrease of f / u molar reports. In other words, the degree of branching of the hardened UF resin decreased with the decrease of F / U Molar reports. Figure 11 shows that the glazing time of the UF resins increased and the ground loss decreased when the molar ratio f / u decreased by 1.60 to 0.95. This indicates that the reactivity and the degree of condensation reaction has decreased with the slowdown of F / U Molar reports during the polymerization process. In other words, the influence of the condensation reaction decreased in hardened resin and that of crystallization increases with the decrease of F / U Molar reports. Figure 12 shows Thea TPA at peak 2, when UF resins with different Molar ratios f / u were dried in conditions of different pH values. When the molar ratio f / u gradually decreased from 1.60 to 0.95, the variations OFA TPA with the decrease of pH passed from the decrease in increase. When the molar ratio f / u was 1.05, the OFA TPA variation with pH values was very limited. This result indicates that the turning point of the importance of condensation or crystallization during the hardening of the resin was about the molar ratio f / u of 1.05. When the molar ratio f / u was greater than 1.05, the condensation reaction played an important role. However, when the molar ratio f / u was less than 1.05, the crystallization played an important role. Fig. 9.A peak 2 on DSC curves of UF resin with different f / U polymerization molar reports with pH 3.0 fig. 10.a X-ray diffractograms of hardened resins hardened with different f / u molar reports at pH 3.0 Fig. 11.A Gel Time and the mass loss corresponding to the peak 2 of UF resin with different f / u ratios hardening molars in conditions of pH 3.0 fig. 12.a tpa peak 2 on DSC curves of UF resin with different / U Molar ratios f of hardening in conditions of different conclusions pH condensation reaction, and as well as crystallization takes place during the resin polymerization process (UF). When the resin is undurated with a high molar ratio f / u, the condensation reaction takes place before the aggregation of the macromolecular chains of initial resin. The reaction increases the degree of ramification of resin and weakens the crystallization. On the contrary, when the resin is undurated with a lower molar ratio, aggregation and crystallization crystallization Chains take place before the condensation reaction. The reaction imposes the crystallinity of the hardened resin. With the decrease of F / U Molar reports, the importance of the condensation reaction and the importance of crystallization gradually convert each other during the polymerization process. When the molar ratio f / u is higher than 1.05, the condensation reaction plays an important role. When the molar ratio f / u is less than 1.05, crystallization plays an important role. The turning point is around a molar ratio of 1.05. The molar ratio, and as well as the pH condition, will influence the polymerization process through the condensation reactivity of the initial resin. The impact of the molar ratio on the resin polymerization is greater than the impact of the pH condition. This indicates that the effect of hardener on hardened resin properties is limited. References Cited Dazmiri, M. K., Kiamahalleh, M. V., Dorieh, A., and Pizzi, A. (2019). Effect Ã ¢ of the initial / u Molar ratio F in Urea-formaldehyde resins synthesis and its influence on medium-density fiber performance linked with them, Ã ¢ Ã, International Journal of Accession and adhesives, 102440. Doi: 10.1016 / J . ijadhadh.2019.102440 Ding, ZJ, and Tian, â €