



大连理工大学
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Exploration on the improvement and application of 2D resin

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1. Background

2D Resin

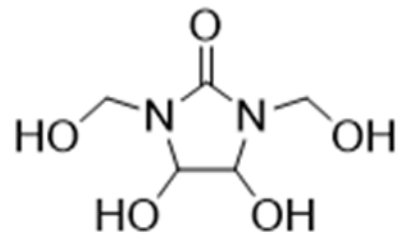


Figure1.1 2D Resin

2D resin, the full name of dihydroxymethyl dihydroxyethylene urea (DMDHEU), has excellent hydrolysis resistance, good storage stability, good washing ability, and is widely used in fabric shaping.



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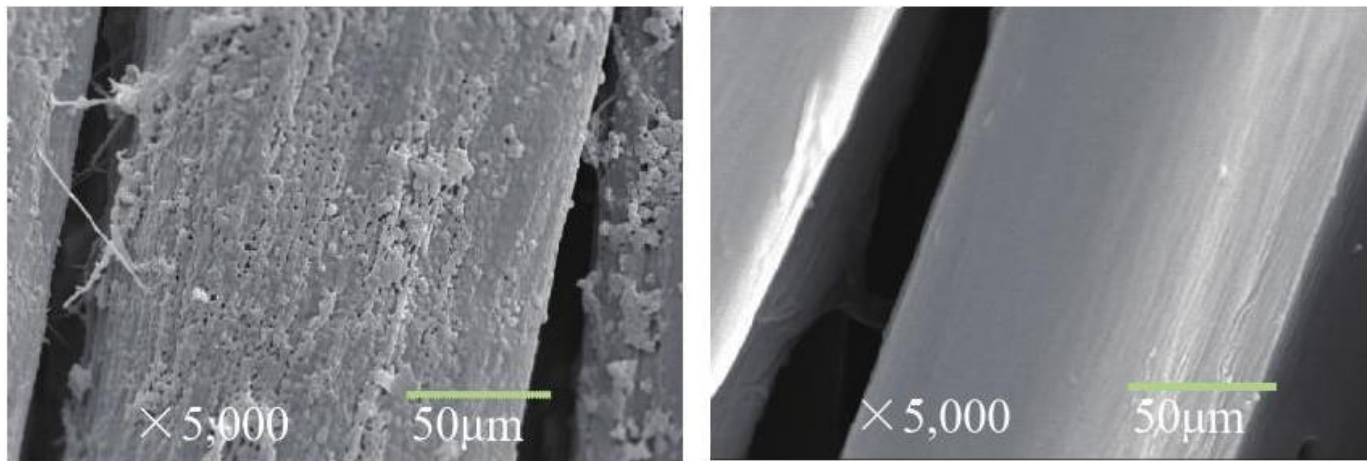


Figure1.2 Before(left) and after(right) adding 2D resin ^[1]

DMDHEU with fiber crosslinking reaction, inside the fiber to form stable mesh structure, make the fiber macromolecule between mobile and sliding, so the fabric in the fiber treated by resin position is relatively stable, which seldom happened under the water swelling large displacement, scaling effect, so it in cotton and its blended fabric has **good durability and anti-wrinkle effect**.

[1] 韩国军, et al. 2D树脂对平纹织物抗皱整理的因素分析. 山东纺织科技, 2010, 51(02): 8-10.



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Defects in 2D resin synthesis process

In the process of finishing, storage and wearing, 2D resin will release **formaldehyde** which is harmful to the human body and the environment. Meanwhile, high temperature finishing under acidic conditions will degrade cotton fibers, resulting in the loss of tensile strength, tearing strength and **wear resistance** of fabrics^[2]. Therefore, the improvement of 2D resin has always been the focus of attention.

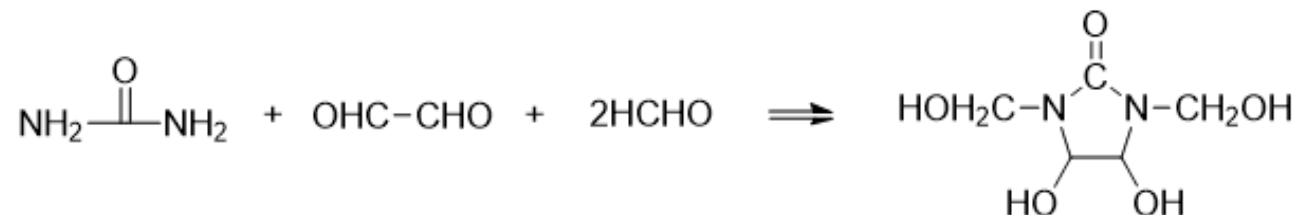


Figure1.3 Synthesis reaction diagram of 2D resin

[2] 陈新福, 张玉高. 棉织物的双丙烯酸酯类碱性无甲醛交联整理[J]. 印染, 2019, 45(17): 26-30.



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At the beginning of the project, it is planned to synthesize a modified version of 2D resin based on melamine from 2D resin and melamine which have been industrially produced :

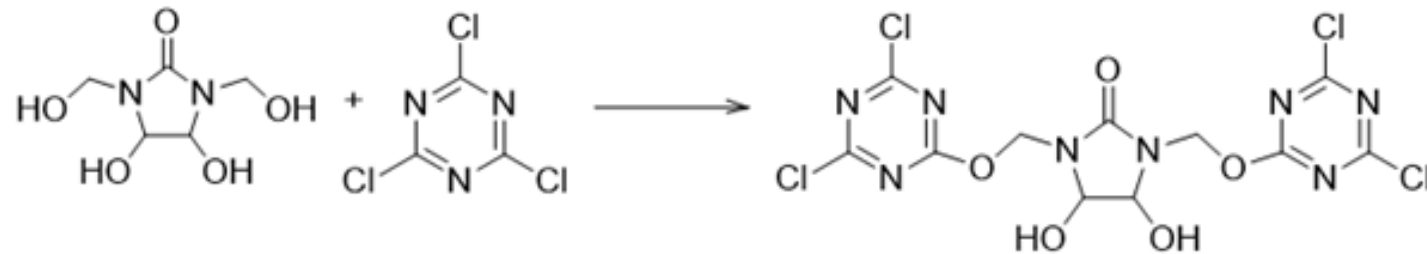


figure2.1 Synthesis reaction of 2D resin
based on modified version of melamine



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The initial 4, 5-dihydroxyimidazoline-2-ketone (DHEU) of 2D resin was intended to be used for cross-linking reaction with melamine. DHEU was synthesized by the scheme shown in FIG. 2.2, and it was confirmed by ^1H NMR spectrum analysis that DHEU was obtained, but the yield was quite low. DHEU was only slightly soluble in DMSO.

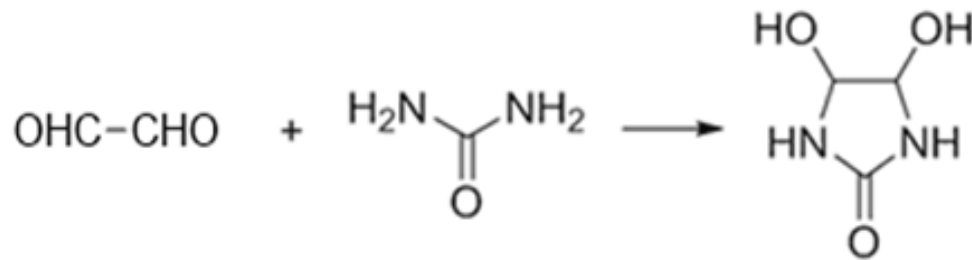


figure2.2 Schematic diagram of DHEU synthesis reaction



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Schematic diagram of DHEU synthesis reaction A modified version of DHEU based on melamine was attempted by vigorously stirring DHEU with melamine in DMSO and DMF.

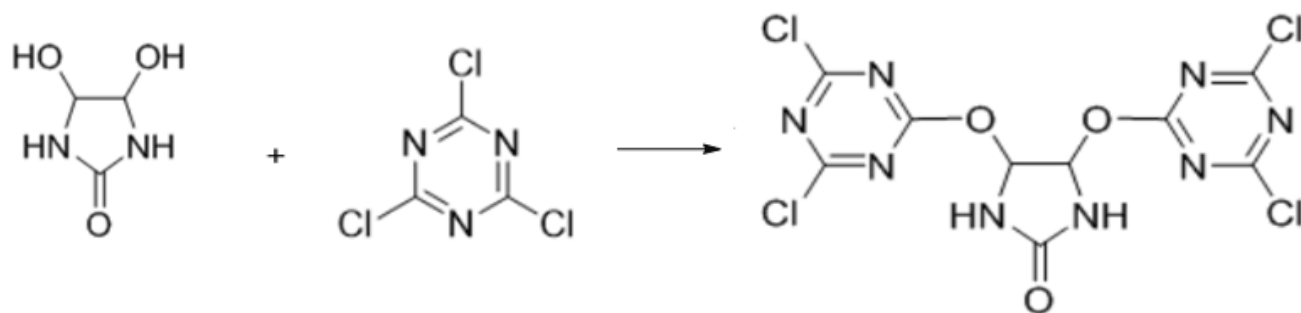


figure2.3 Reaction diagram of DHEU synthesis based on melamine modified version



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Instead of DHEU, we turned to 2, 3-dihydroxypropanamide, both amides, and both hydroxyl groups can be used to cross-link with melamine. We used the literature method [2] [3] [4] as shown in Figure 2.4 to try to synthesize 2, 3-dihydroxypropanamide by oxidizing acrylamide with potassium permanganate.

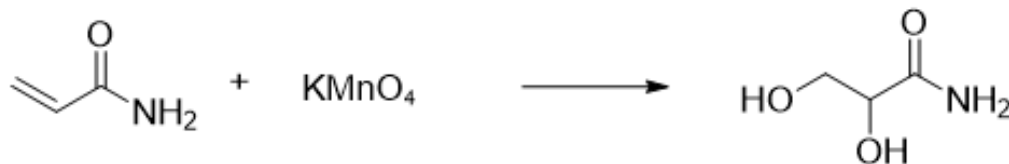


figure2.4 Schematic diagram of synthesis reaction of 2, 3-dihydroxypropanamide

- [3] Drudis-Sole, G.; Ujaque, G.; Maseras, F.; Lledos, A., *Chemistry-a European Journal* **2005**, 11, 1017.
- [4] Dotsenko, V. V.; Aniskina, Y. E.; Strelkov, V. D.; Dyadyuchenko, L. V.; Krapivin, G. D. *In Oxirane-2,2-dicarboxamides: synthesis, reactions and biological activity*, 20th International Electronic Conference on Synthetic Organic Chemistry, Electr Network, Nov 01-30; Electr Network, 2016.
- [5] Pilichowski, J. F.; Mailhot, G.; Bolte, M., *Organic Preparations and Procedures International* **1994**, 26, 551.



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Acrylamide (3.0 g, 42.2 mmol) was dissolved in water (8 mL) and acetone (5 mL), and the solution was prepared. The solution was prepared with acetone (60 mL), water (40 mL), and potassium permanganate (7.33 g, 46.4 mmol) and placed in an ice salt bath at -10 °C, to which the acrylamide solution was slowly added, and the solution was finished in half an hour. The ice salt bath was removed, the mixture was stopped after stirring for 30 min, and the white solid i was obtained after filtration and concentration.



figure2.4 Schematic diagram of synthesis reaction of 2, 3-dihydroxypropanamide



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^1H NMR data showed in Figure 2.5 that about 1/4 of the acrylamide was oxidized to the target product, 2, 3-dihydroxypropanamide.

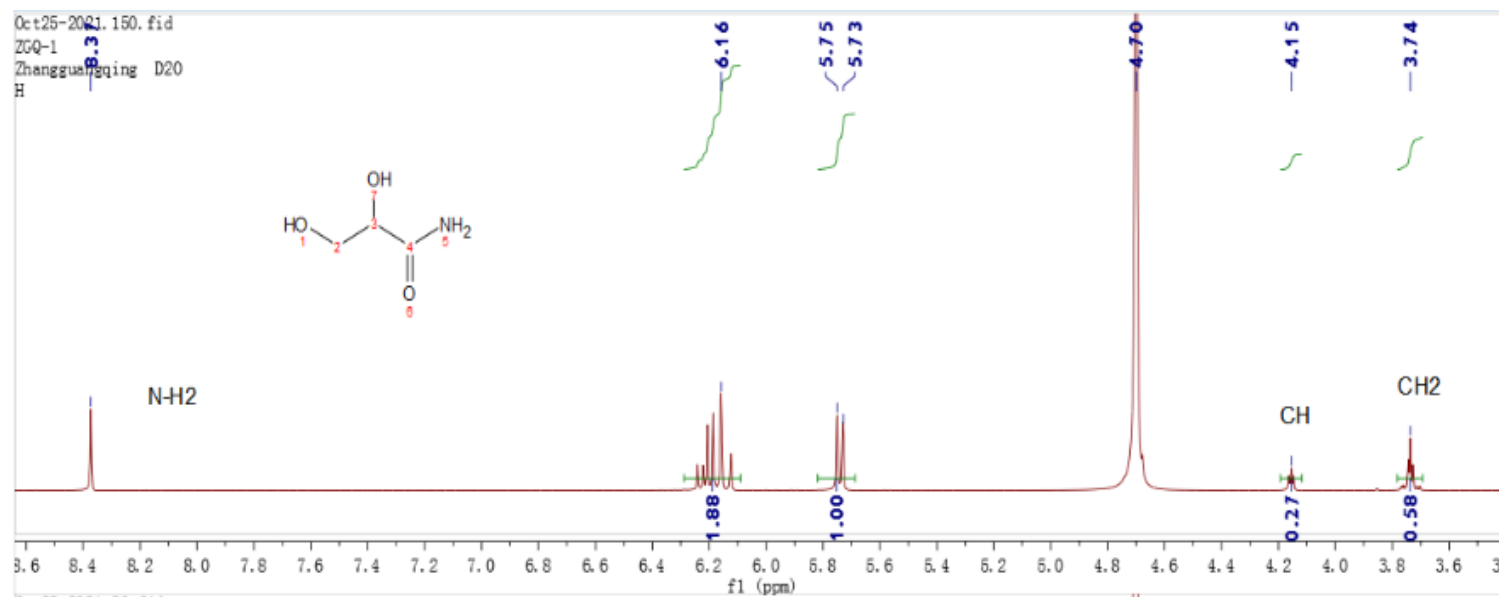


figure2.5 ^1H NMR spectra of solid III



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The mixture containing 2, 3-dihydroxypropanamide was tried to react with melamine in order to synthesize the modified version of 2, 3-dihydroxypropanamide based on melamine. The reaction formula is shown in FIG. 2.6 .

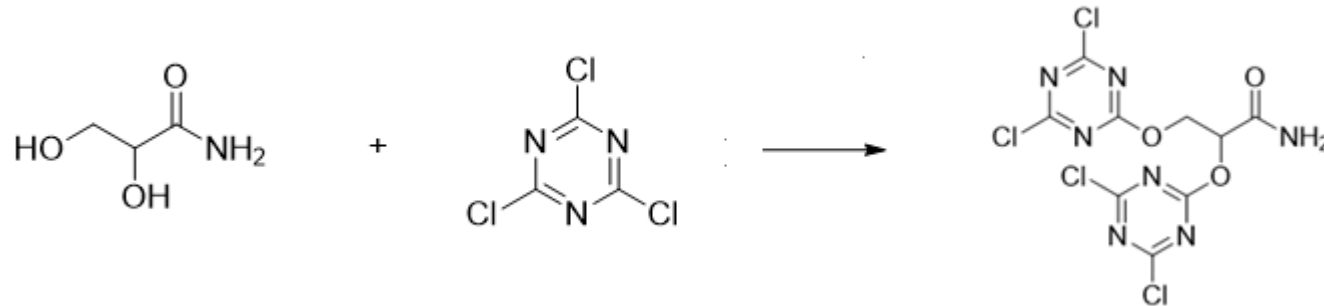


figure2. 6 Schematic diagram of synthesis reaction of 2, 3-dihydroxypropanamide based on melamine modified version



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White solid i (including part of 2, 3-dihydroxypropanamide) was tried to dissolve with 3 mL DMF, and it was found that the dissolution effect was very bad, and it was still cloudy after 2 minutes of ultrasound. After adding melamine and stirring overnight, it was still found that TLC could not detect the reaction process .

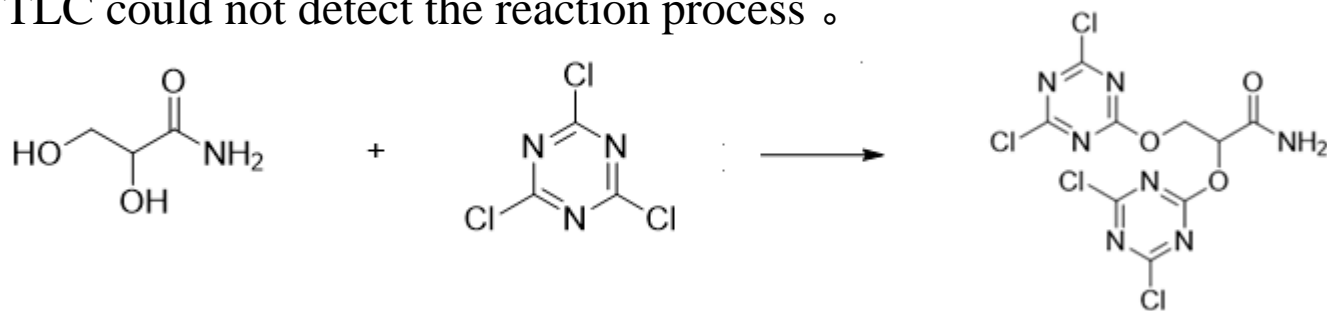


figure2.6 Schematic diagram of synthesis reaction of 2, 3-dihydroxypropanamide based on melamine modified version



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Project Output :

The cross-linking reaction of synthetic DHEU with melamine was explored. 2, 3-dihydroxypropanamide was synthesized from acrylamide and its reaction with melamine was explored.

Results outlook :

Although the exploration based on melamine has not been successful due to the problem of the solubility of raw materials and products, it provides an important reference for the research and development of fabric shaping agents. For example, the cross-linking of polyethylene glycol with melamine with better solubility can be considered for further exploration.



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- [1] 韩国军, et al. 2D树脂对平纹织物抗皱整理的因素分析. 山东纺织科技, 2010, 51(02): 8-10.
- [2] 陈新福, 张玉高. 棉织物的双丙烯酸酯类碱性无甲醛交联整理[J]. 印染, 2019, 45(17): 26-30.
- [3] Drudis-Sole, G.; Ujaque, G.; Maseras, F.; Lledos, A., *Chemistry-a European Journal* **2005**, 11, 1017.
- [4] Dotsenko, V. V.; Aniskina, Y. E.; Strelkov, V. D.; Dyadyuchenko, L. V.; Krapivin, G. D. *In Oxirane-2,2-dicarboxamides: synthesis, reactions and biological activity*, 20th International Electronic Conference on Synthetic Organic Chemistry, Electr Network, Nov 01-30; Electr Network, 2016.
- [5] Pilichowski, J. F.; Mailhot, G.; Bolte, M., *Organic Preparations and Procedures International* **1994**, 26, 551.



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THANKS
FOR LISTENING