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Original Research Article

Study on Preparation and Modification of Hydrazodicarbonamide

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ABSTRACT

This study relates to an improved method for they preparation of hydrazodicarbonamide, an intermediate in the preparation of azodicarbonamide which is useful as a chemical blowing agent. Hydrazodicarbonamide is prepared by reaction of urea and hydrazine in acidic medium. Hydrazine is a relatively expensive chemical and this is reflected in the cost of materials derived from it. Accordingly, any means of effecting a substantial increase in the yield of hydazodicarbonamide from hydrazine is commercially very important. By Series of comparative experiments, Factors of influencing the yield of biurea which is prepared from the condensation reaction between hydrazine hydrate and urea are studied.

Keywords: Azodicarbonamide, Hydrazodicarbonamide, Blowing agent, Hydrazine, Urea.

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Introduction

Polymer foams in the modern world are widely used in the manufacture of foams in the manufacture of furniture, insulation materials, sponges, etc. [1, 2]. Foams are produced by mixing the solid phase with the gas. The gaseous phase is caused by a puffy reagent that can produce gas physically or chemically [3, 4]. In physical foaming agents, gas is caused by physical changes such as evaporation, but in chemical foaming agents, gas is produced by a chemical reaction such as thermal decomposition [5]. Azodicarbonamide (ADCA) with the chemical formula $NH_2CONNCONH_2$ is the most well-known chemical foaming agent for the preparation of polymeric foams [6], which is produced through the oxidation of hydrazodicarbonamide (HADC) in the presence of an oxidizer [7, 8].

HDAC is a white solid of the molecular formula $NH_2CONHNHCONH_2$ and is prepared from the reaction of urea with hydrazine or its salts. The HDCA decomposes at 230-260 ° C and produces gas, a white solid and a viscous liquid. The gas produced contains ammonia and carbon dioxide. The white solid is urea and viscous liquid is urazol [9]. Various methods have been proposed in the literature for HDCA synthesis. Some of these steps are summarized as follows:

1. Reaction of hydrazine or its salts with urea in the presence of non-oxidizing acids such as sulfuric acid, phosphoric acid or hydrochloric acid [10-15].

2. Reaction of hydrazine or its salts with urea in the presence of an alkali and the removal of ammonia produced from the reaction mixture [16].

3. The reaction of a ketazine with urea in the presence of a non-oxidizing acid [17].

4. The reaction of a ketazine with urea in the presence of an alkali produced ammonia separation and the unhydrolyzed ketone and ketazin from the reaction mixture [18].

5. Urea reaction with semicarbazide [19].

In this paper, the factors affecting the synthesis of HDCA were investigated using the first method.

Experimental

Generel

A mixture of 200 parts of monohydrazine sulfate, 270 parts of technical urea and 300 parts of water was heated to the refluxing temperature under refluxing conditions and with agitation. After about two hours of boiling the pH had risen to 4.0 whereupon a total of 63 parts of commercial concentrated sulfuric acid was added dropwise at such a rate as to hold the pH at a level between 2 and 2.5. After two- and three-quarter hours of additional boiling the acid addition was discontinued and the pH of the mixture was allowed to rise while the mixture was boiled for a further period of fifteen minutes. The reaction was discontinued at the end of five hours total elapsed time. The reaction mixture was then cooled and filtered and the filtered precipitate was washed.

Results and discussion

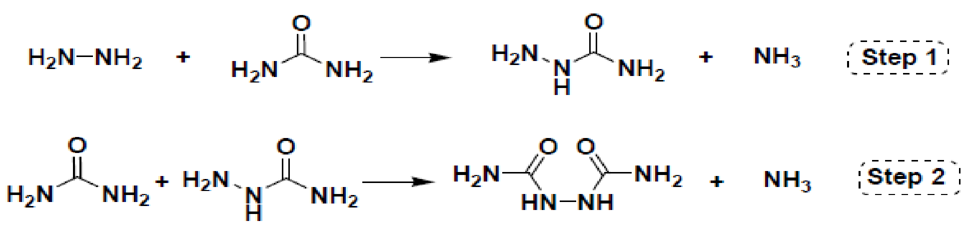
The influence of pH: During the hydrazine preparation process, large amounts of sodium carbonate, sodium hydroxide, and sodium chloride are produced that prevent the substitution reaction between urea and hydrazine. Therefore, sodium carbonate and sodium hydroxide must be removed from the reaction medium. By cooling the hydrazine solution before the urea is increased, the sodium carbonate dehydrates into a precipitate, and much of it is dissolved in the solution. The remainder of the acid exits during reflux in the form of carbon dioxide gas and the gain is neutralized. According to the laboratory method, sulfuric acid is used as a neutralizer, you can see the results of pH effect in Table 1.

Table 1. Effect of pH on the yield of HADC

Entry	experiment	Yield[a] (%)
1	Increase acid in one step	55
2	Increase acid in two step	85

[a]Isolated yield.

As shown in Table 1, in the first experiment only acid was added at the beginning of the reaction which gave a reaction yield of 55%, whereas in the second experiment the acid was added again after 4 h of reflux to maintain the pH and yield increased. This is a two-step reaction. In the first step, an intermediate called semicarbazide is formed and in the second step, HDCA is produced. The reaction steps are as follows:



It is necessary to consider the temperature and pH that affect the type of product, for example, if T <30 ° C and pH <1, hydrazine sulfate is formed. If the temperature is 50 ° C <T<90 ° C and 7<pH <9, semicarbazide is formed and HDCA is formed if 85 °C< T <95 ° C and 2 <pH <6 [9].

The effect of N_2 gas: As shown in Figure 1, if the reaction is carried out under nitrogen gas during reflux, the yield of HDCA will increase.

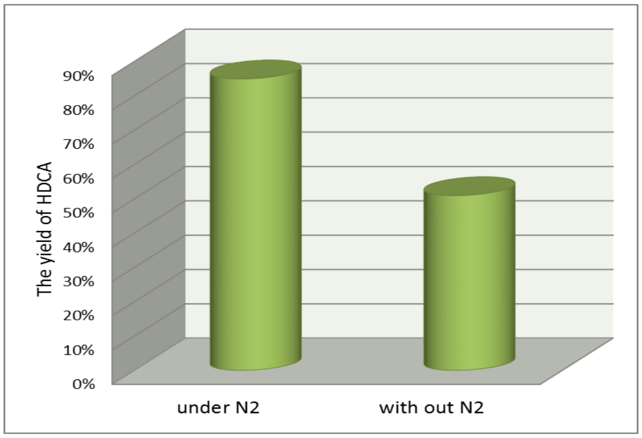


Figure 1. The effect of N_2 gas on the yield of HADC

Since at high temperature hydrazine reacts with oxygen in the following reaction, it exits the reaction medium [20]. Therefore, using inert gas can prevent this reaction and thereby increase the yield of reaction.

$N_2H_4 + O_2 \rightarrow N_2 + 2H_2O$

Conclusion

Since hydrazine is an expensive material, many efforts have been made to increase the yield of hydrazodicarbonamide production and further increase azodicarbonamide. The results of this study show that adding the right amount of acid and performing the reaction under gas have a good effect on increasing the yield of HADC**.**

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