Synthesis of GChMA and Its Infrared Spectroscopic Analysis

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Annotation: in this study, guanidine rhodanide, guanidine chloride, methanal, and acrylic acid were used to synthesize guanidine chloride methyl acrylate (GChMA). The optimal conditions for the synthesis process were established, and the influence of temperature on the reaction was examined. The molecular structure of the synthesized compound was analyzed using infrared (IR) spectroscopy, and the results confirmed that the obtained structure was in full agreement with the theoretical predictions.

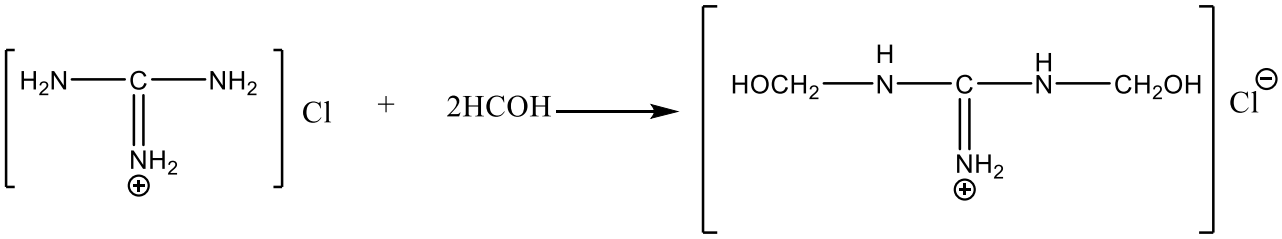
Key words: formaldehyde, acrylic acid, guanidinium chloride, guanidinium chloride methyl acrylate (GChMA), IR spectroscopy, guanidine rhodanide

For the synthesis of an effective corrosion inhibitor, guanidine, methanal (using 38-40% formalin as the methanal source), and acrylic acid were selected as the target substances. To conduct the reaction under laboratory conditions, a 500 mL three-necked flask was used. The three-necked flask was equipped with a thermometer, and a magnetic stirrer was installed to ensure proper mixing of the solution in the flask. Guanidine was extracted from the waste of NavoiAzot JSC, which contained guanidine rhodanide. Concentrated hydrochloric acid was added to this solution to obtain guanidine chloride. The resulting mixture was then filtered to isolate the final product. Upon analyzing the filtrate, the presence of rhodanide acid was identified. To isolate the rhodanide acid, a solution of $CuCl_2$ was added until the precipitation process was complete. The resulting mixture was then filtered and purified. To examine the solution containing guanidine chloride, it was evaporated, and the guanidine chloride was isolated [1; 2].

The obtained guanidine chloride was dissolved in water, and its molecular mass was determined using the cryoscopic method. Additionally, IR spectroscopy results were obtained and compared with standard references. The purified guanidine chloride was redissolved in water to prepare a solution, which was then transferred to a three-necked flask for further synthesis[3-5].

During the synthesis, the reaction kinetics were studied by considering key influencing factors, including concentration, temperature, and reaction duration. The reaction between guanidine chloride, obtained from guanidine rhodanide, and methanal proceeds in two stages. In the first stage, the monomethylol derivative of guanidine chloride is formed. In the second stage, due to the excess ratio of methanal, the dimethylol derivative is formed. In this derivative, the number of donor atoms increases; however, the solubility of the substance decreases, leading to the formation of a pale yellow precipitate.

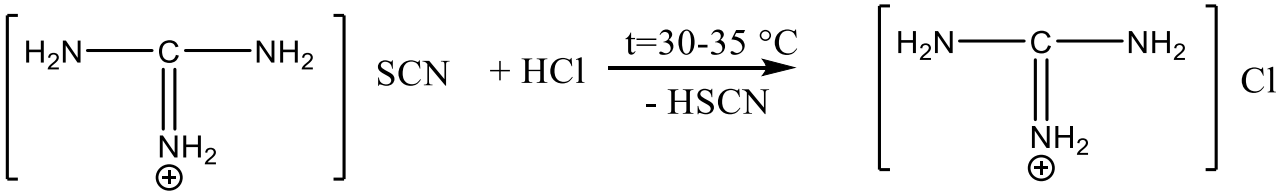
To prevent the formation of the dimethylol derivative, it was necessary to mix guanidine chloride and methanal in a 1:1 ratio. To ensure the precise execution of the process, it was considered appropriate to use an excess amount of guanidine chloride.



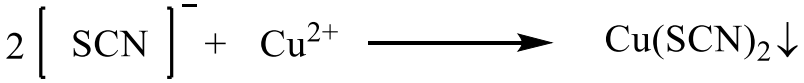
Because the solubility of the synthesized dimethylol guanidine derivative differs from that of the monomethylol derivative, controlling the reaction conditions is crucial to obtaining the desired product.

Effect of Temperature on the Synthesis Process

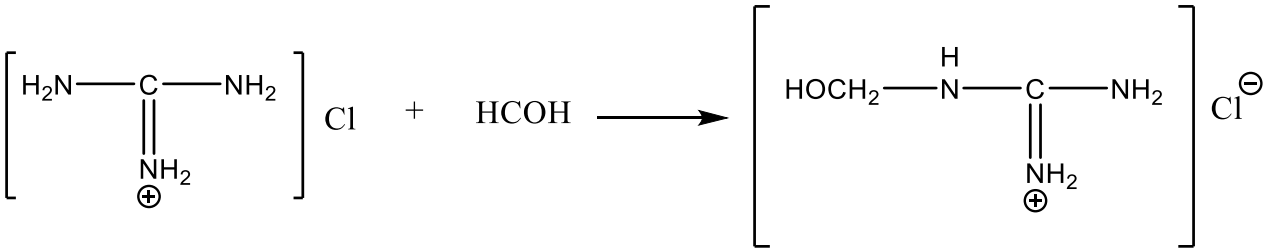
The effect of temperature on the synthesis process based on guanidine, methanal, and acrylic acid was investigated. Below, the sequential reaction equations of the process are provided.



To obtain guanidine chloride from guanidine rhodanide, the reaction was carried out at a temperature range of 30-35°C.



The isolated guanidine chloride was mixed with a methanal solution in a 1:1 molar ratio in a 500 mL three-necked flask and heated to 50-60°C. As a result, a yellowish methylol derivative of guanidine chloride was obtained.



At each stage, the isolated substances were analyzed using mass spectrometry (MS) and infrared (IR) spectroscopy. The obtained product was designated as GChMM (guanidine chloride monomethylol). The synthesized GChMM was reacted with acrylic acid in a 1:1 molar ratio at a temperature of 70-75°C, resulting in the synthesis of GChMA (guanidine chloride methyl acrylate). The reaction yield of the first and second stages was determined by conducting experiments at different temperatures, as presented in Table 1.

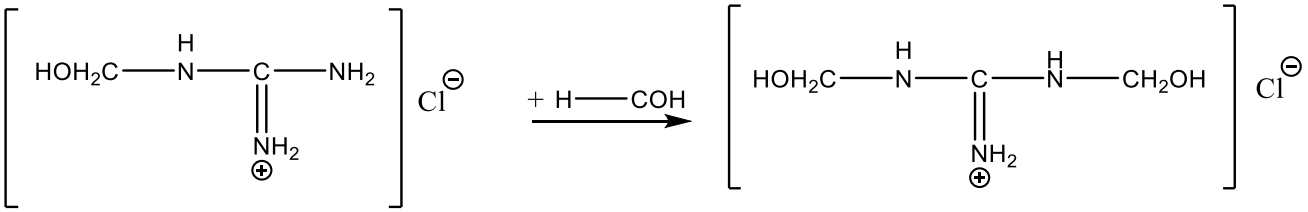
Table 1

Dependence of GXMM Yield on Temperature

№	Temperature of reaction, °C	Yield of reaction, %
1	30	44,3
2	40	48,5
3	45	59,6
4	50	78,1
5	55	80,5
6	60	83,1
7	70	82,3

When the reaction temperature was 30°C, the reaction yield was 44.3%, while at 40°C, it increased to 48.5%, and at 45°C, it reached 59.6%. Within the 50-60°C range, the reaction yield was between 78.1% and 83.1%.

However, when the temperature exceeded 60°C and reached 70°C, a decrease in yield was observed. This was explained by the reduced solubility of methanal in the reaction mixture and the formation of guanidine chloride dimethylol. If the temperature exceeds 60°C, an additional reaction occurs, leading to the formation of the following compound.



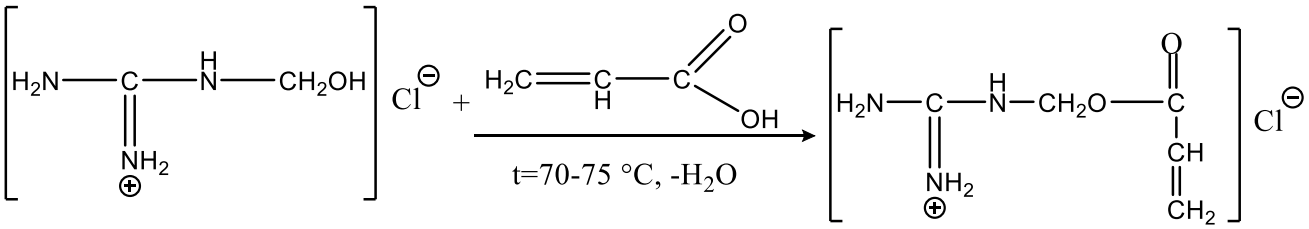
The synthesized GChMM was isolated and subsequently reacted with acrylic acid in the next stage. In this process, the maximum reaction yield of 86.5% was achieved within the 70-75°C temperature range.

Table 2

Dependence of GXMA Yield on Temperature

№	Temperature of reaction, °C	Yield of reaction, %
1	35	48,2
2	48	50,3
3	55	58,6
4	60	75,8
5	70	83,8
6	75	86,5
7	80	81,3

The reaction equation for the formation of GChMA can be expressed as follows:



As the reaction mixture temperature increases, the reaction yield decreases significantly. The primary reason for this is the reduction in the reaction between acrylic acid and the methylol group, leading to a reverse reaction, i.e., de-etherification. To enhance the reaction rate, concentrated (96%) sulfuric acid was used as a catalyst. A mixture of GChMM and acrylic acid in a 1:1 molar ratio was treated with 5 mL of concentrated sulfuric acid and heated within the 70-75°C range.

As a result of the reaction, the formation of GChMA was indicated by the appearance of a denser mass in the solution. The formation of this compound was confirmed using various physicochemical analytical methods, and the relevant conclusions were drawn (Figure 1).

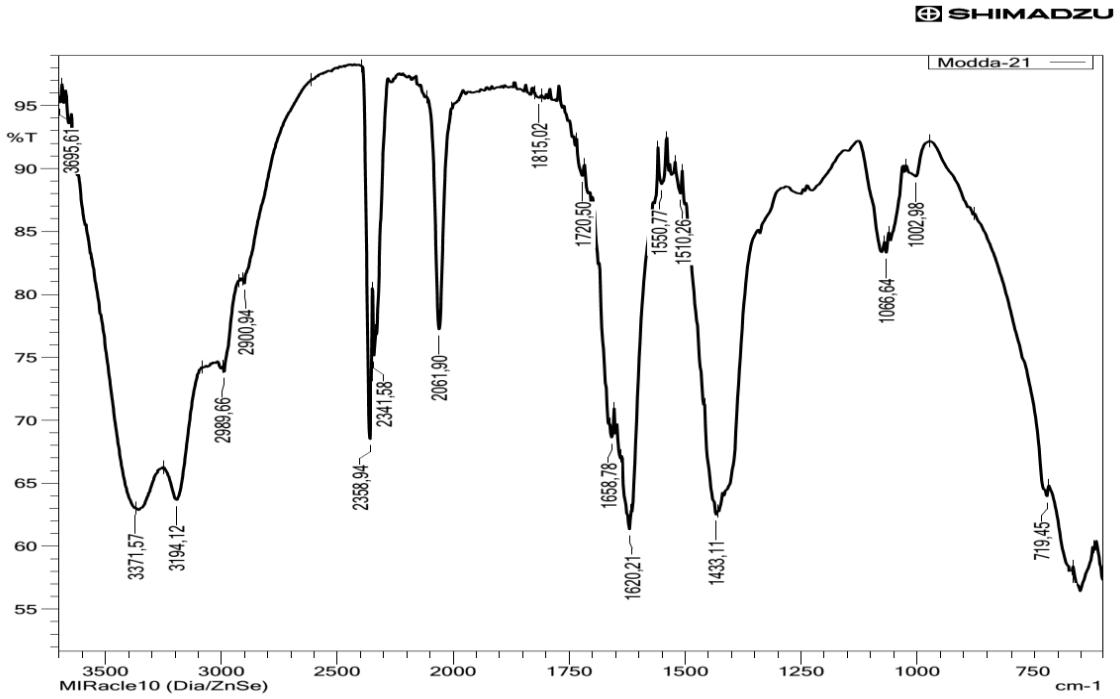


Figure 1: Infrared Spectroscopy Analysis of Synthesized GChMA

The obtained analytical data revealed characteristic absorption bands at 3371.57 cm^{-1}, 3194.12 cm^{-1}, 2989.66 cm^{-1}, 2358.94 cm^{-1}, 2061.90 cm^{-1}, 1720.50 cm^{-1}, 1658.78 cm^{-1}, 1620.21 cm^{-1}, and 1433.11 cm^{-1} in the IR spectrum.

- The absorption at 3371.57 cm^{-1} corresponds to the –C=NH (imine group), confirming its presence.

- The band at 3194.12 cm^{-1} is characteristic of $–NH_2$ (amine group) stretching vibrations.

- The absorption at 2061.90 cm^{-1} indicates the presence of the –C=C– (alkene group).

- The band at 1658.78 cm^{-1} is associated with –C=O (carbonyl group) stretching vibrations.

- Additionally, absorptions observed in the 1625–1725 cm^{-1} range, particularly the 1720.50 cm^{-1} intense band, correspond to valence vibrations of the –C=N group in the guanidine molecule. The Fourier-transform infrared spectroscopy (FTIR) absorption spectrum provides strong scientific evidence that the synthesized product is GChMA (a guanidine-based compound). These absorption bands confirm the presence of various functional groups, demonstrating their formation during the synthesis process.

Thus, the conducted analysis confirms that the synthesized GChMA product possesses the expected chemical structure. A comparison between the IR spectrum of guanidine chloride from the spectral database and the newly synthesized

compound reveals new functional group-specific absorption bands. This confirms that the synthesized GChMA product is fully consistent with the reaction of guanidine, methanal, and acrylic acid.

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