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SYNTHESIS, STRUCTURAL CHARACTERIZATION, AND COLLOIDAL AND SURFACE-ACTIVE PROPERTIES OF A SURFACTANT BASED ON MONOETHANOLAMINE-MODIFIED GOSSYPOL RESIN INVESTIGATED BY FTIR AND PHYSICOCHEMICAL METHODS

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Abstract

In this study, a surfactant was obtained through the chemical modification of gossypol resin, formed as a by-product of cottonseed oil processing, with monoethanolamine, and its structural and surface-active properties were comprehensively investigated. The modification process was carried out stepwise in an alkaline medium, and the effect of reaction parameters on product yield was analyzed to determine the optimal conditions. The structural changes of the obtained product were analyzed using FTIR spectroscopy. The appearance of new absorption bands in the 1200–1300 cm^{-1} region indicated the formation of C–N bonds and confirmed the chemical incorporation of monoethanolamine into the gossypol structure.

The surface-active characteristics of the synthesized material were studied using surface tension, critical micelle concentration, foam formation, and foam stability. It was found that the material is capable of lowering surface tension, forming micelles, and forming stable foam. These characteristics point to its possible use as a flotation agent for the enrichment of coal, as well as for the extraction of valuable elements from coal processing wastes.

Keywords: *Gossypol resin, monoethanolamine, chemical modification, FTIR spectroscopy, surfactant, surface tension, critical micelle concentration, foaming properties, amphiphilic structure*

Introduction

In modern chemical technologies, the efficient utilization of industrial waste and the production of high-value products are among the key priority areas. In particular, gossypol resin, formed as a by-product during cottonseed oil processing, accumulates in significant amounts, and its rational utilization remains an important research challenge. This waste contains reactive functional groups such as phenolic, carbonyl, and hydroxyl groups, which make it a promising raw material for obtaining new functional materials through chemical modification.

In recent years, numerous studies have focused on the synthesis of surfactants based on natural and renewable raw materials, as well as on the investigation of their physicochemical properties. Such approaches enable the development of environmentally friendly, cost-effective, and biodegradable surfactants. In this context, the modification of natural resins and phenolic polymers with amine-containing compounds to form amphiphilic structures is considered a promising direction. However, the production of efficient surfactants through the modification of gossypol resin with amine reagents, particularly monoethanolamine, has not yet been sufficiently and systematically studied.

Gossypol resin possesses a chemical structure that enables it to react with compounds containing amino groups. As a result of such modification, a balance between hydrophilic and hydrophobic fragments is formed within the molecule. This enhances the adsorption capacity of the substance at the solution–air interface, reduces surface tension, and promotes micelle formation. Therefore, the modification of gossypol resin with monoethanolamine to obtain new types of surfactants is of considerable scientific and practical importance.

In this study, the modification of gossypol resin with monoethanolamine was investigated, and the optimal reaction conditions were determined. The structural characteristics of the obtained product were analyzed using FTIR spectroscopy, confirming the formation of new chemical bonds as a result of modification. In addition, the surface-active properties of the product were evaluated in terms of surface tension, critical micelle concentration, and foaming ability, demonstrating its potential for practical applications.

However, the production of surfactants through the modification of gossypol resin with monoethanolamine has not been sufficiently investigated.

This approach has not been previously reported. The aim of this work is to obtain a surfactant through the chemical modification of gossypol resin with monoethanolamine, to determine its structural characteristics using FTIR spectroscopy, and to comprehensively evaluate its surface-active properties by physicochemical methods.

Research method

In this experimental study, gossypol resin, formed as a by-product during cottonseed oil processing, was used as the main raw material. The sample was obtained from a local industrial enterprise, dried under laboratory conditions, and brought to a homogeneous dispersed state. Monoethanolamine (MEA) was used as a modifying reagent, while sodium hydroxide (NaOH) was applied to provide an alkaline medium. All reagents used in the study were of analytical grade and were used without further purification.

Table 1. *Synthesis Conditions and Results of Monoethanolamine Modification of Gossypol Resin*

No.	MEA Content (wt.%)	Temperature (°C)	Time (h)	Catalyst (NaOH, %)	Ratio (GR: MEA)	Product Yield (%)	Remarks
1	10	75	2	1.0	100:10	65	Low conversion
2	20	75	2	1.0	100:20	70	Reaction initiation
3	30	75	2	1.0	100:30	68	Excess amine effect
4	10	75	4	1.0	100:10	72	Moderate yield

No.	MEA Content (wt.%)	Temperature (°C)	Time (h)	Catalyst (NaOH, %)	Ratio (GR: MEA)	Product Yield (%)	Remarks
5	20	75	4	1.0	100:20	82	Maximum yield
6	30	75	4	1.0	100:30	78	Side reactions
7	10	75	6	1.0	100:10	70	Stabilization stage
8	20	75	6	1.0	100:20	80	Optimal range
9	30	75	6	1.0	100:30	76	Decline phase

The modification of gossypol resin with monoethanolamine was carried out in two stages. Initially, the raw material was hydrolyzed in an alkaline medium at a temperature of 70–85 °C to generate active reaction centers. The obtained hydrolysate was then reacted with monoethanolamine at concentrations of 10–30 wt.% at 70–80 °C for 2–6 hours. The process was conducted under continuous stirring. After completion of the reaction, the product was neutralized, separated, and dried.

The amount of monoethanolamine, reaction temperature, and reaction time were selected as the main parameters of the modification process. The effect of these parameters on the product yield was evaluated based on experimental data, and the optimal conditions were determined.

Based on the data presented in Table 1, the optimal conditions were identified as 20 wt.% monoethanolamine and a reaction time of 4 hours.

The structural characteristics of the obtained product were investigated using FTIR spectroscopy. The spectra were recorded in the range of 4000–400 cm^{-1}, and the functional groups and structural changes resulting from the modification were analyzed.

The surface-active properties of the product were evaluated in terms of surface tension, critical micelle concentration, as well as foaming ability and stability. Surface tension was measured at different concentrations, and the critical micelle concentration was determined from its dependence on concentration. Foaming properties were studied using a mechanical foaming method, and both foam volume and its stability over time were determined.

The results were presented in the form of graphs and tables, and the effect of modification conditions on the product properties was analyzed in detail.

Results and Discussion

The efficiency of gossypol resin modification with monoethanolamine was evaluated based on the effect of reaction parameters on product yield. The amount of monoethanolamine, reaction temperature, and reaction time were selected as the main factors of the study. The obtained results showed that the product yield increases with increasing reagent amount up to a certain limit; however, excessive amounts lead to a decrease in efficiency due to side reactions.

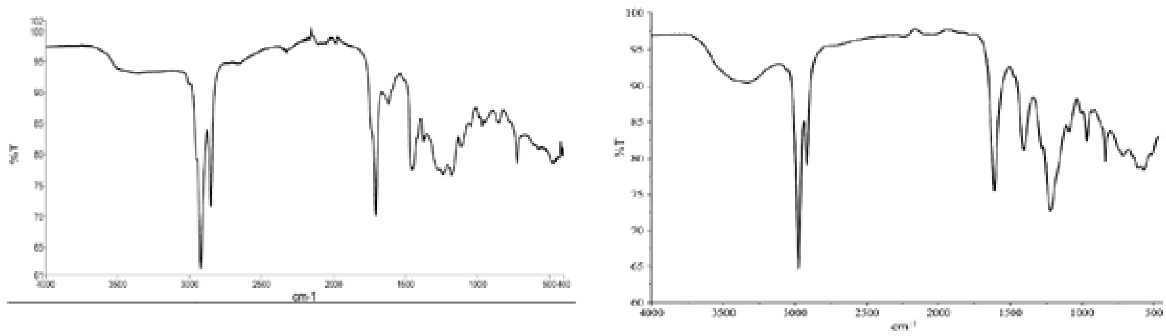
According to the data presented in Table 1, the maximum product yield was observed at 20 wt.% monoethanolamine and a reaction time of 4 hours. This result can be explained by the optimal participation of active centers in the reaction. The effect of reaction temperature on product yield was also investigated separately. An increase in temperature accelerates the reaction; however, above a certain temperature, side reactions become more pronounced, as shown in Figure 1.

The effect of reaction time on surface-active properties was further studied. As the reaction time increases, the degree of modification also increases, leading to an improvement in surface-active properties; however, longer reaction times result in higher energy consumption. The results shown in Figure 2 indicate that the decrease in surface tension confirms the effectiveness of the modification process.

The effect of monoethanolamine content on foaming properties was also investigated. The maximum foaming ability was observed at an optimal concentration. The structural changes of the obtained product were determined using FTIR spectroscopy. The spectra of samples before and after modification were compared.

Figure 1. *FTIR spectra of gossypol resin: (a) unmodified, (b) modified with monoethanolamine*

a) b)

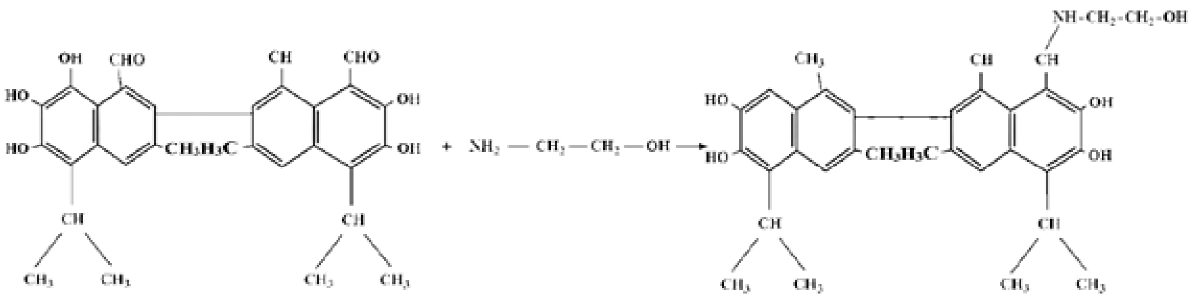


The FTIR spectra of gossypol resin are presented in Figure 1. In the spectrum of the unmodified sample (Figure 1 a), a broad absorption band observed in the range of 3200–3500 cm^{-1} is characteristic of hydroxyl (O–H) groups. The absorption peaks at 1600–1650 cm^{-1} indicate the presence of carbonyl (C=O) groups, while the bands in the range of 1000–1200 cm^{-1} correspond to C–O bonds.

In the modified sample (Figure 1 b), the appearance of new absorption bands in the range of 1200–1300 cm^{-1} confirms the formation of C–N bonds, indicating the chemical incorporation of monoethanolamine into the gossypol structure. At the same time, the retention of absorption bands corresponding to O–H and C=O groups indicates that the main structure of the material was preserved during modification. The emergence of new bands in the fingerprint region further indicates an increase in molecular structural complexity.

These results demonstrate that the reaction between gossypol resin and monoethanolamine leads to significant changes in the molecular structure. During the reaction, functional groups interact with monoethanolamine molecules, resulting in the formation of new chemical bonds.

Figure 2. *Reaction scheme of gossypol resin modification with monoethanolamine*



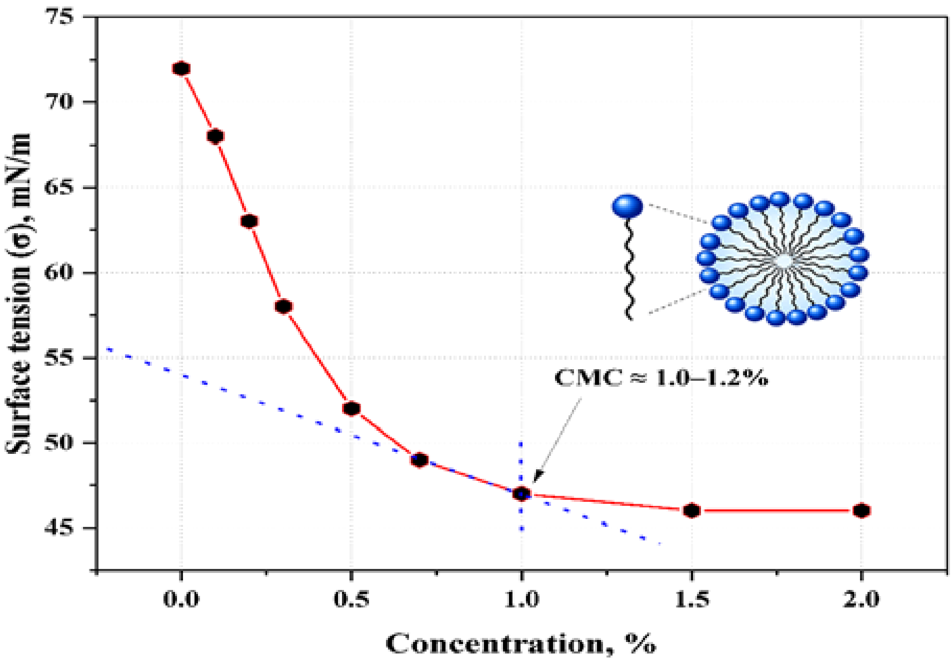
As a result, an amphiphilic structure consisting of hydrophilic and hydrophobic fragments is formed in the molecule. This plays an important role in the formation of the surface-active properties of the material and determines its potential for practical applications.

The formed amphiphilic molecules spontaneously aggregate in an aqueous medium to form micellar structures, which ensure the manifestation of the surface-active properties of the material. In this case, hydrophilic groups interact with the surrounding medium, while hydrophobic fragments are located in the inner part of the micelle.

One of the key properties of a surfactant is its ability to reduce surface tension. Experimental results showed that as the concentration of the substance increases, a sharp decrease in surface tension is observed.

According to the results shown in Figure 3, as the concentration increases up to 1.0%, the surface tension decreases sharply and then gradually stabilizes. This confirms the presence of the critical micelle concentration (CMC).

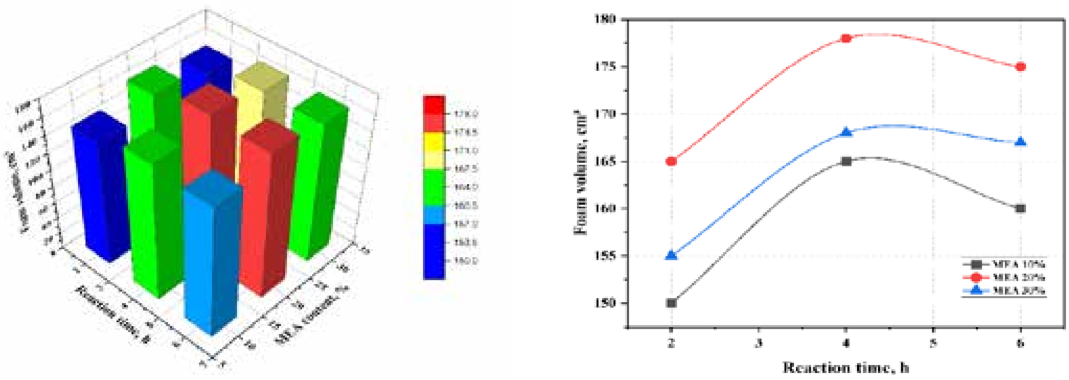
Figure 3. *Dependence of surface tension on concentration*



The foaming properties of the modified gossypol-based surfactant were evaluated under different reaction conditions. According to the obtained results, the monoethanolamine content and reaction time have a significant effect on the foaming properties.

Figure 4. *Effect of monoethanolamine content and reaction time on foaming properties: (a) 3D plot, (b) 2D plot*

a) b)



According to the results presented in Figure 4, the maximum foam volume reached 178 cm³ and was observed at a monoethanolamine content of 20% and a reaction time of 4 hours. These conditions were identified as optimal. The obtained results allowed the main performance indicators of the material to be summarized in tabular form.

According to the data presented in Table 2, the obtained material reduces surface tension to 46–47 mN/m, confirming its effectiveness as a surfactant. The high values of foam formation and stability further substantiate the potential of the material for application as a surfactant.

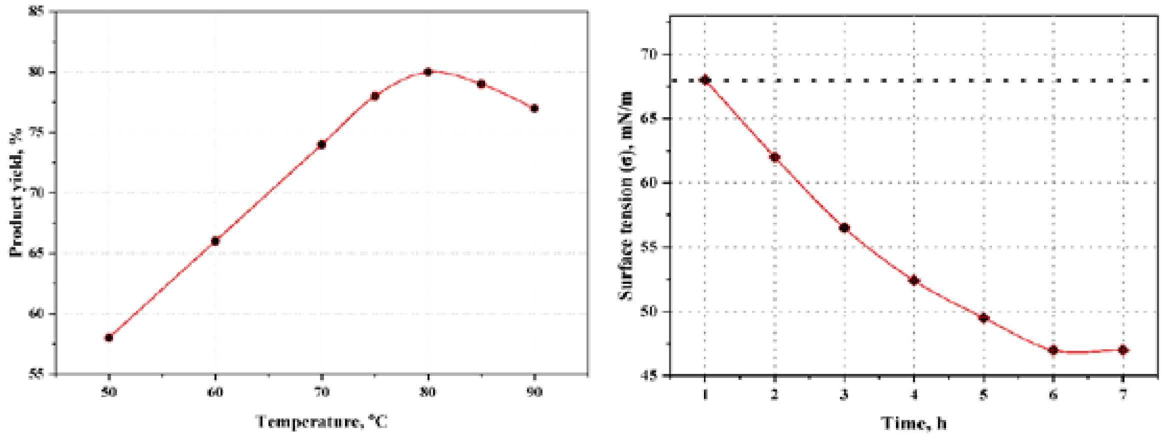
The effect of reaction parameters on the surface-active properties was comprehensively analyzed. During the modification with monoethanolamine, the reaction conditions, including temperature, time, and reagent amount, significantly influence the physicochemical properties of the product.

Table 2. *Main properties of the modified gossypol-based surfactant*

Parameter	Value	Description
Surface tension, mN/m	46–47	Minimum value after the critical micelle concentration (CMC)
Foam volume, cm^3	178	Under optimal conditions (20% monoethanolamine, 4 h)
Foam stability, %	70–75	Stability over 5–10 minutes

Figure 5. *Effect of reaction parameters on the surface-active properties of gossypol resin during modification with monoethanolamine: (a) effect of temperature on product yield, (b) effect of reaction time on surface tension*

a) b)



According to the results shown in Figure 5(a), as the reaction temperature increases, the product yield initially increases and reaches a maximum value (≈82%) at around 80 °C. Further increase in temperature leads to a decrease in product yield due to the intensification of side reactions and partial thermal degradation. This confirms the existence of an optimal temperature range for the modification process.

Figure 6. *Effect of monoethanolamine content on the foaming properties of the modified gossypol-based surfactant*

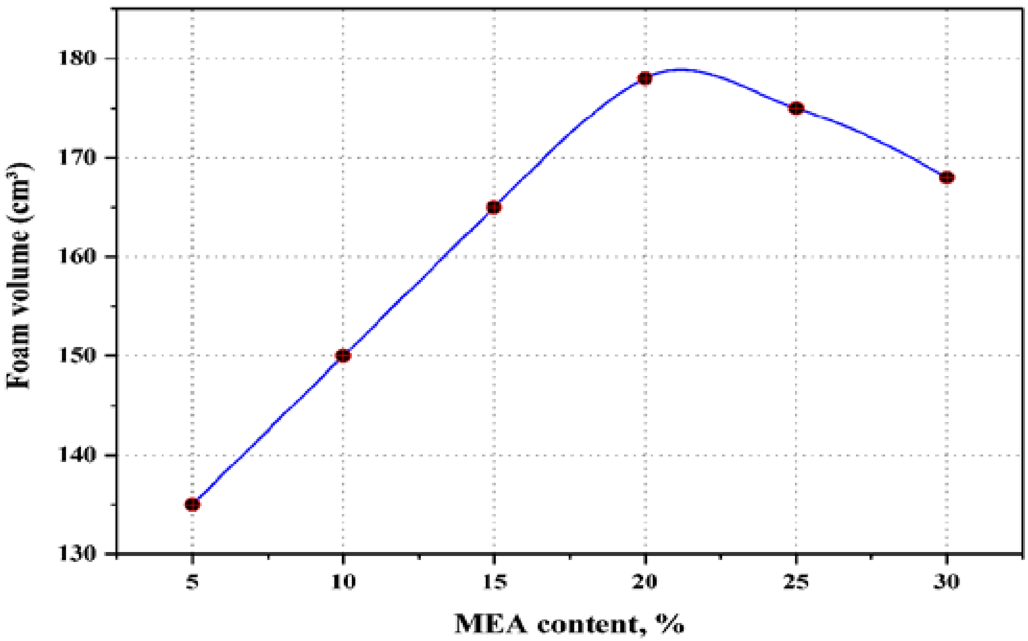


Figure 5(b) presents the effect of reaction time on surface tension. According to the obtained data, as the reaction time increases, the surface tension decreases and reaches a minimum value (≈46–47 mN/m) within the range of 5–6 hours. This indicates that the modification process approaches completion and that the surface-active properties have been established.

As the monoethanolamine content increases, the foam volume increases and reaches a maximum value (≈178 cm^3) at 20%. Beyond this value, the decrease in foam volume is attributed to the disruption of the balance between hydrophilic and hydrophobic fragments.

Conclusion

In this study, a surfactant was obtained through the chemical modification of gossypol resin with monoethanolamine, and its structural and surface-active properties were comprehensively investigated. The FTIR analysis results indicate the formation of C–N bonds in the 1200–1300 cm^{-1} range during the modification process and confirm the chemical incorporation of monoethanolamine into the gossypol structure.

The effect of reaction parameters on product yield was studied, and the optimal conditions were determined to be 20% monoethanolamine content and a reaction time of 4 hours, under which the product yield reached approximately 82%. Analysis of the surface-active properties showed that the material reduces surface tension to 46–47 mN/m and that the critical micelle concentration lies within the range of 1.0–1.2%.

In addition, the material exhibited high foaming ability (≈178 cm^3) and sufficient foam stability (70–75%), confirming its potential application as an effective surfactant. Compared with conventional surfactants, the obtained material demonstrates competitive surface-active performance and favorable physicochemical properties.

The obtained results demonstrate that the development of new surfactants based on gossypol resin and the efficient utilization of industrial waste represent a promising direction.

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