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COMBINATION OF GAMBIER EXTRACT AND BENZOIC ACID AS INHIBITOR OF CALCIUM SULFATE SCALE FORMATION

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ABSTRACT

In this study, it was carried out the addition of a mixture of gambier (*Uncaria gambir* Roxb.) extract and benzoic acid as an inhibitor of scale growth of calcium sulfate using the seeded experiment technique. The experiment was performed on variations in the concentration of calcium sulfate growth solution with variation from 0.15 to 0.25 M by the presence of inhibitor mixture 0 - 125 ppm and a temperature of 90 ° C. Observations on the growth of calcium sulfate crystals formed were carried out by weighing crystals obtained in each series of experiments. Characterization of calcium sulfate crystals obtained was done by particle size analyzer/PSA and scanning electron microscope/SEM. The results obtained showed that the addition of a mixture of gambier extract and benzoic acid at a ratio of 1: 1 may block the formation of calcium sulfate crystals with effectiveness in the range of 2 - 54% depending on the concentration of the inhibitor added. The results obtained are supported by data obtained by SEM and PSA analysis. The data obtained from SEM and PSA confirmed that the size of calcium sulfate crystals after the addition of the inhibitor becomes to be smaller than the normal growth.

Keywords: Scaling Inhibitor, Calcium Sulfate, Gambier Extract, Benzoic Acid, Green Inhibitor.

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INTRODUCTION

The use of natural materials to overcome human life problems related to environmental problems has recently been a concern of researchers.¹⁻¹⁰ The use of natural materials such as plant extracts, algal biomass, fungi, activated carbon, and cassava peel have been carried out by researchers as a corrosion inhibitor and an adsorbent for heavy metals and dyes that pollute the environment.¹¹⁻³⁵ Extract of gambier and kemenyan were also used as inhibitors of inorganic material scale formation.^{36,37} The use of natural materials aims to overcome problems in the industrial world without having to cause new problems for the environment.

Gambier (*Uncaria gambir* Roxb.) extract and modified gambier extract are known as natural materials that promise to inhibit the growth of calcium carbonate (CaCO₃) and calcium sulfate (CaSO₄) scale.³⁶⁻³⁸ Modification of gambier extract with several types of organic acids was reported to be able to block the CaCO₃ crystal growth.³⁹ However, the use of gambier extract modified with benzoic acid as an inhibitor for the growth of CaSO₄ crystals is still unknown. Modification of gambier extract in the presence of organic acids such as citric acid or benzoic acid was conducted to control the gambier extract quality.³⁹

Noting the potency of the gambier extract modified as an effective inhibitor of $CaCO_3$ scale growth, in this study it was reported that the use of gambier (*Uncaria gambir* Roxb.) extract modified with benzoic acid as an inhibitor of $CaSO_4$ scale growth. The addition of benzoic acid was carried out to overcome the damage of gambier extract from microorganisms so that it can be stored for a long time. The experiments were conducted using the seeded experiment method by measuring alteration in the weight of $CaSO_4$ crystals produced either without inhibitors or by adding inhibitors. The obtained crystal identification was formed with a particle size analyzer (PSA) and scanning electron microscope (SEM) to prove that inhibitors work in blocking the $CaSO_4$ crystal growth.

EXPERIMENTAL

Materials and Instruments

The instruments used in this study consisted of water baths, plastic bottles, magnetic stirrers, analytic balance (Airshwoth AA-160), Scanning electron microscopy (JEOL jsm-6510, Japan), particle size analyzer (Coulter LS 100Q, United State of America).

The materials used in this study included acetone, benzoic acid, anhydrous calcium chloride, sodium sulfate, aquabidest, acetone, distilled water, filter paper, gambier (*Uncaria gambir* Roxb.) extract obtained from Padang-West Sumatera and Palembang (South Sumatera) gambier plants. The chemicals were bought from Merck-Germany.

Gambier Extract Preparation

Extraction of gambier was prepared by smoothing gambier solids (Padang and Palembang). An amount of 100 grams of gambier in the form of powder was placed in 1 L of distilled water. The gambier solution is heated at 80 ° C and stirred using a stirrer for 2-3 hours. The solution was filtered to obtain a concentration of gambier extract in 100000 ppm. This method was repeated in the preparation of 100000 ppm benzoic acid solution. The two solutions were filtered and mixed with a ratio of 1: 1.

Seed Crystals Production

Seed crystals were made by blending 1 M anhydrous CaCl₂ solution and 1 M Na₂SO₄ solution in 50 mL aquabidest respectively. These compounds were heated and stirred with a magnetic stirrer at 80 °C for 30 minutes. The mixture was left at room temperature to get seed crystals. The obtained crystals were filtered and washed with cold aquabidest and acetone, continued with drying in an oven at a temperature of 105 °C. This technique was replicated at certain times until a sufficient number of seed crystals were obtained to carry out the experiments.

Inhibitor Activity Test

Determination of the Growth of CaSO₄ by Variations in Growth Media Concentration without the Presence of Inhibitor

The growth solutions were produced from blending of $0.15 \text{ M CaCl}_2 (200 \text{ mL})$ and $\text{Na}_2\text{SO}_4 (200 \text{ mL})$ into a 500 mL plastic bottle. The mixture was stirred for 15 minutes at 90 °C and then placed into the water bath for 15 minutes at 90 °C to reach equilibrium. Then the mixture was poured into 7 plastic bottles contained in the water bath at 90 °C each with 50 mL followed by the addition of 200 mg CaSO₄ crystal seeds into plastic bottles at the same time. Every 5 minutes, the precipitate formed in the bottle is removed, filtered with filter paper, and washed with acetone and then dried at 105 C in the oven. The crystals formed were weighed and they were characterized by SEM and PSA. This experiment was repeated in a variation of the CaSO₄ growth media concentration of 0.20 - 0.25 M.

Determination of CaSO₄ Crystal Growth by Variations in Growth Media Concentration with the Addition of Inhibitor

The procedure performed in this experiment was the same as the procedure for determining the CaSO₄ growth rate without the presence of inhibitors. But in this experiment, the addition of inhibitors was carried out simultaneously in a solution of CaCl₂ and Na₂SO₄ before the two solutions were mixed. The inhibitors used were a mixture of gambier extract and benzoic acid solution with a ratio of 1: 1. The inhibitor concentration was added 0, 25, 75, and 125 ppm in the CaSO₄ growth media concentration of 0.15, 0.20, and 0.25 M. An amount of CaSO₄ crystal seeds added was made to remain as much as 200 mg. The precipitate formed by the addition of inhibitors was weighed then analyzed by SEM and PSA. The data obtained in the form of the amount of precipitate to time with variations in the concentration of growth solutions and inhibitors, each was plotted as the weight of deposit to time by Microsoft Excel 2013. The slope value obtained from each graph is the CaSO₄ scale growth rate. Changes in crystal size or changes in CaSO₄ crystal morphology before and after the addition of inhibitors were analyzed by SEM. While changes in the crystal size distribution of CaSO₄ before and after the addition of inhibitors were analyzed by SEM.

RESULTS AND DISCUSSION

Gambier extract is very susceptible to microorganisms and fungi. The quality of gambier extract at room temperature does not last more than 3 days. The addition of benzoic acid to gambier extract with a ratio of GAMBIER EXTRACT AND BENZOIC ACID AS INHIBITORS 142 Subarso *et al.*

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1: 1 can improve the quality of mixed extract. This can be proven from the observation that the addition of benzoic acid increases the quality of the mixture. It can last for more than 2 months. The mixture of gambier extract and benzoic acid was then used as an inhibitor to block the CaSO₄ crystals growth. The graph of changes in the weight of CaSO₄ crystal precipitate to time in various variations of CaSO₄ growth solution concentration is shown in Fig.-1. The growth solution of CaSO₄ was made in the concentration range of 0.15 to 0.25 M. In this concentration range it is possible to observe the growth of CaSO₄ crystals and measure the weight gain of the crystals produced. From the data in Fig.-1 it can be observed that the growth of CaSO₄ crystals at higher concentrations takes place faster. In this case, the growth of CaSO₄ crystals at a 0.25 M growth media takes place faster than the growth media concentration by 0.20 and 0.15 M as indicated in Fig.-1. The fast growth rate of CaSO₄ crystals is evidenced by the increase in the amount of $CaSO_4$ crystals formed. The amount of $CaSO_4$ crystals formed in the final state of observation in the volume of 1 L solution with the growth media concentration of 0.25 M approached 14 g. While the amount of crystals formed at the growth media concentrations of 0.20 and 0.15 M are in the range of 11 and 7 g. In general, the increase in the amount of deposits formed from the material inorganic is immediately comparable to the rise in the concentration of the growth solution of the material. This is in line with some of the experiment results from previous works as reported by researchers on calcium phosphate material ⁴⁰ and on calcium carbonate and sulfate materials.^{41,42}



Fig.-1: Weight Changes of CaSO4 Crystals at Different Growth Media Concentrations

The influence of the inhibitor addition from the gambier extract and benzoic acid mixture on a range of $CaSO_4$ growth media concentrations may be identified in Figs.-2, 3, and 4. In these figures it may be observed that the addition of a mixture of gambier extract and benzoic acid can impede the $CaSO_4$ crystal growth. Increasing the amount of mixture of gambier extract and benzoic acid into growth solutions increases the inhibitor's effectiveness in blocking the $CaSO_4$ crystals growth. But when the concentration of the growth solution was made higher, the inhibitor ability decreased in blocking the $CaSO_4$ crystals growth. This is due to the higher the concentration of $CaSO_4$ growth solution (0.25 M), $CaSO_4$ crystal growth takes place faster than the growth media concentration of 0.15 and 0.20 M which results in reduced inhibitor work. This also occurs in the addition of tobacco extract in blocking the growth of $CaCO_3$ and $CaSO_4$ crystals.⁴³

The inhibitors effectiveness in blocking $CaSO_4$ scale formation can be measured by the percent effectiveness of inhibitors and calculated using Eqn.-1.^{3,44}

Effectiveness of inhibitors (EI %) =
$$\frac{(Wa - Wb)}{(Wo - Wb)} \times 100$$
 (1)

Where, W_a is the weight of the precipitate with the addition of inhibitors at equilibrium (g) in volume 1 L, W_b is the weight of the precipitate without the addition of inhibitors at equilibrium (g), W_o = initial weight (g) of seed crystals added into the growth media in volume 1 L.

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The results of the calculation of the inhibitor's effectiveness (%) in a mixture of gambier extract and benzoic acid in blocking the growth of CaSO₄ crystals at inhibitor concentrations 0 - 125 ppm and concentrations of CaSO₄ growth solution by 0.15 - 0.25 ppm are displayed in Table-1. From Table 1 it can be observed that the ability of the mixture of gambier extract and benzoic acid in blocking the formation of CaSO₄ crystals in the range of 2 - 54%. The highest ability of the mixture of gambier extract and benzoic acid in inhibiting CaSO₄ scale formation (54%) takes place at the CaSO₄ concentration (0.15 M) by the volume of inhibitors appended as much as 125 ppm. While the lowest ability of the mixture of gambier extract and benzoic acid in blocking the formation of CaSO₄ scale (2%) takes place at the CaSO₄ growth solution concentration of the amount of inhibitors added as much as 25 ppm. The addition of the mixture of gambier extract and benzoic acid solution as much as 125 ppm followed by an increase in the CaSO₄ growth solution concentration from 0.15 to 0.25 M resulted in the decreased inhibitory ability of this inhibitor. This is also shown in the addition of kemenyan extract in blocking the formation of calcium carbonate crystals.³⁷ A similar case was also found in the presence of poly (citric acid) to the growth of CaSO₄ crystals.²



Fig.-2: Changes in CaSO₄ Crystal Weight at A Concentration of the CaSO₄ (0.15 M) with Variations in the Concentration of Addition of Inhibitors

To prove visually the inhibitor work in blocking the CaSO₄ crystals growth, the CaSO₄ crystals formed without the addition of inhibitors and with the addition of inhibitors were observed by a scanning electron microscope. The data of the analysis with SEM are described in Fig.-5. In Fig.-5 it may be observed that the morphology of CaSO₄ crystals, in general, has not changed. However, the CaSO₄ crystal size by the presence of inhibitors makes smaller and more fragile textures.

Deeper observations on the CaSO₄ crystal size with and without the appearance of inhibitors were carried out taking a particle size analyzer (PSA). The results of the analysis of CaSO₄ crystal size without and with the addition of inhibitors may be referred to in Fig.-6. Figure-6 represents that the presence of inhibitors inside the growth media of CaSO₄ changes the particle size distribution of CaSO₄ that grows. The shift in the particle size distribution of CaSO₄ by the appearance of inhibitors occurred with the lower particle size distribution compared to the addition of inhibitors. The average diameter of CaSO₄ particle without the addition of inhibitors is around 20 m μ and after the addition of inhibitors, the particle diameter of CaSO₄ becomes around 10 m μ (Fig.-6).

The existence of gambier extract and benzoic acid solutions containing chemical compounds such as tannins, catechins, quercetin, and benzoic acid serve as inhibitors to block the CaSO₄ scale growth.^{36,37} The mechanism of inhibitor inhibition in blocking the growth of the CaSO₄ scale in the case of this study can be through the threshold inhibition mechanism. In the threshold inhibition mechanism, the solubility of CaSO₄ due to the presence of compounds contained in gambier and benzoic acid can increase the solubility of CaSO₄ so that the supersaturation as a condition for crystallization to be reduced. The mechanism of complex formation between Ca²⁺ metal ions and functional groups found in chemical compounds contained

in gambier and benzoic acid such as carboxylates can also occur. The complex formation between the Ca^{2+} metal ions and carboxylic groups inhibits the formation of a reaction between ions of Ca^{2+} and SO_4^{2-} so that the growth or formation of CaSO₄ crystals becomes slowed.



Fig.-3: Changes in CaSO₄ Crystal Weight at A Concentration of CaSO₄ (0.20 M) by Variations in the Concentration of Inhibitor Addition



Fig.-4: Changes in CaSO₄ Crystal Weight at A Concentration of CaSO₄ (0.25 M) with Variations in the Concentration of Inhibitor Addition

Table-1: Effectiveness of Inhibitors with the Growth Solution Concentration Range

Concentrations of CaSO4 Growth Solution					
0.15 (M)		0.20 (M)		0.25 (M)	
Inhibitor	Inhibitor	Inhibitor	Inhibitor	Inhibitor	Inhibitor
Concentration	Effectiveness	Concentration	Effectiveness	Concentration	Effectiveness
(ppm)	(%)	(ppm)	(%)	(ppm)	(%)
0	0.00	0	0.00	0	0.00
25	31.99	25	2.41	25	2.43
75	41.13	75	5.53	75	5.73
125	53.76	125	23.97	125	13.47

The presence of tannins in gambier also has a contribution to the crystal dispersion mechanism. The tannin compound which is a macromolecular compound can absorb the surface of CaSO₄ crystals derived from crystalline seeds added to the growth solution as a result of CaSO₄ crystals being dispersed in small sizes as shown in Fig. 6. The mechanism of dispersion and the formation of complexes on the growth of CaSO₄

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crystals was also common also of poly (citric acid) which can inhibit the growth of CaSO₄ crystals with inhibitory effectiveness exceeding 90%.² The mechanism of dispersion also occurred in blocking the formation of calcium phosphate crystal by the presence of poly (acrylic acid).⁴⁵ The results of this work are in line with the inhibition of growth of CaSO₄ crystals in the addition of mixed phosphonate inhibitors ⁴⁶ and modified gambier with kemenyan.⁴⁷ The results of this work also may be contrasted with another inhibitor as liquid smoke to block the growth rate of calcium carbonate.⁴⁸ Thus it can be stated that the addition of a mixture of gambier extracts and benzoic acid by a comparison of 1 : 1 to the growth solution of CaSO₄ can inhibit the growth of CaSO₄ scale.



Fig -5: Morphology of CaSO₄ Crystals (a) without and (b) with the Addition of Inhibitors





CONCLUSION

The addition of inhibitor mixture extract gambier and benzoic acid at a ratio of 1:1 can block the CaSO₄ crystals growth with the effectiveness of the inhibitor of 2 - 54% in the concentration of CaSO₄ growth media by 0.15-0.25 M. The effectiveness of inhibitors depends on the CaSO₄ growth media concentration and added inhibitor. The inhibitor effectiveness was 54% in the CaSO₄ growth solution concentration of 0.15 M by the addition of 125 ppm inhibitor. The effectiveness of inhibitor so of inhibitors will decrease when the growth media concentration is increased from 0.15 M and the added inhibitor concentration is lower than 125 ppm. The data of the characterization by SEM and PSA confirmed that the morphology and diameter size of CaSO₄ crystals were smaller with the inhibitor addition of gambier extract and benzoic acid mixture.

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