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Microwave-assisted extraction of eco-friendly surfactant from *Jatropha curcas* for sustainable solubilization of reactive dyes

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Abstract

Natural surfactants derived from plant-based sources, such as saponins, remain underexplored. This study developed the extraction of saponins from *Jatropha curcas* leaves using microwave-assisted extraction (MAE) finding that the optimized condition of 3 min, 363.15 K, 30 mL/g ratio of extraction yielded the highest saponin content of 35.04 mg/g. The FTIR and HPLC analyses confirmed the structural similarity between the extract and commercial saponin. Additionally, the extracted saponins effectively solubilized Remazol Red RB and Blue TQ with solubilization efficiency increasing proportionally to the surfactant concentration. The surfactant properties of the extracted saponin were also confirmed by its ability to form foam and high critical micellar concentration, which revealed the potential for material valorization. This work demonstrated that the development of plant-based surfactants provides a sustainable alternative to synthetic surfactants. Moreover, valorizing natural materials contributes to the advancement of eco-friendly technologies, particularly in waste treatment and water purification applications.

Keywords: Natural surfactant; critical micelle concentration; surfactant micelles; microwave-assisted extraction

1. Introduction

Surfactants is the main component of detergents, which has an amphiphilic nature. They have been used in many industries such as in oil recovery, emulsification, or as cleaner, wetting agents, and more [1]. However, most commercially available surfactants, like Tween 80 and SDS, are synthetic. Even though synthetic surfactants is economical, it poses a serious environmental challenges, including its hazard for the soil, water and human body if being consumed. It, as a consequence, has made their widespread application limited [2]. Ongoing research, in response, aims to identify environmentally friendly surfactants derived from natural sources.

Natural surfactants obtained from plants are highly desirable for being less toxic, biodegradable, and having greater specificity compared to synthetic alternatives [3]. These surfactants can be extracted from various sources, including animals, microorganisms, and plant parts (seed, fruits, leaves, or flowers), such as in the plant of *Jatropha* [4,5]. Belonging to the *Euphorbiaceae* family, *Jatropha curcas* is one of the primary sources of saponins. Commonly known as physic nut, purging nut, or pig nut, *Jatropha curcas* is widely cultivated in Indonesia and not classified as a food crop [6].

Extracting saponins from *Jatropha curcas* requires an efficient extraction process as several factors determine the extraction yield, including solvent type, solvent concentration, solvent-to-feed ratio, irradiation temperature, extraction duration, and extraction technique [7]. Maceration is a traditional method used to extract non-volatile compounds by immersing raw materials into a solvent. This technique, however, has several drawbacks, such as long extraction times, high solvent consumption, low extraction efficiency, and a high content of inert materials [8]. To address these limitations, modern extraction techniques have been developed to enhance process efficiency, reduce extraction time, increase bioactive compound yields, and minimize energy and solvent consumption. A number of assisted extraction technologies, such as ultrasound, high pressure, and microwaves, offer an improved performance through process intensification mechanisms [9].

Microwave-assisted extraction (MAE) enhances the extraction process by generating heat through microwave radiation, resulting in more rapid energy transfer and uniform heating. This technique offers several advantages, including higher extraction yields, reduced processing time, minimal solvent usage, and cost-effective production of high-quality extracts [10–12]. Additionally, using water as a solvent in MAE aligns with the principles of green chemistry [13].

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Previous studies have primarily focused on the extraction processes and phytochemical properties of natural surfactants derived from plant-based materials [14–17]. However, only a few studies have explored the application of plant-derived surfactants as solubilizing agents. The only study found is when saponin from *Sapindus mukorossi* was applied to solubilize methyl blue and eosin yellow [18], and saponins from *Sapindus rarak* was used for solubilizing water pollutants [2]. For that reason, this study aims to investigate the surfactant and solubilization properties of saponin extracts from *Jatropha curcas* leaves, an area that has not so far been previously explored. Using MAE with water as the solvent, the research examined the surfactant characteristics of the extract and their micellar solubilization properties to trapped dyes. Additionally, to further understand the potential application of saponins as natural surfactants and solubilizing agents, the study evaluated their effectiveness in solubilizing reactive dyes in aqueous systems by determining the solubilization capacity of saponin micelles.

2. Materials and Methods

2.1. Materials

Mature *Jatropha curcas* leaves were procured from the local plantation at Welahan, Jepara, Indonesia. Pure saponin (99%) procured from Sigma Aldrich as the saponin comparative standard. Reactive dye remazol red RB and remazol blue TQ were used to prepare the wastewater model. Meanwhile, the dye was obtained from a local dye manufacturer (Natt Collection, Surakarta, Indonesia).

2.2. Pre-treatment of raw material

Before extraction, the size of dry *Jatropha sp*. leaves was reduced using grinder and sieved to obtain a fine powder (<100 mesh. Then, powder was stored in a jar equipped with silica gel to keep it dry for further applications.

2.3. Extraction of saponin

The dried powder of *Jatropha sp.* leaves was extracted using MAE with the help of a multimode microwave oven (Elektrolux, Indonesia) at the frequency of 2.45 GHz and the maximum output power of 800W. The operating procedure of MAE was adopted from the previous studies [15,19]. The powder of *Jatropha sp.* leaves was solubilized in water solvent at the ratio of 20-60 mL/g. The mixtures were mixed and irradiated at various powers (640, 720, and 800W), temperatures (343.15, 353.15, 363.15, and 373.15K), and times (1,3,6, and 9 minutes) where simple one factor at a time approach was used and total extracted saponin content was taken as the main parameter. The temperature of the MAE apparatus was kept by flowing coolant liquid using pump in and out of the condenser. The extract obtained was then filtered under vacuum condition and evaporated using rotary evaporator (IKA RV 10 Basic) at 303.15K.

2.4. Total saponin compound analysis

The total saponin compound (TSC) was determined by spectrophotometry method [13] based on the absorbance of the aqueous *Jatropha sp*. leaf extract at 276.5 nm using a UV-VIS spectrophotometer. Commercial pure saponin solution at various concentrations was used as a standard to generate a calibration curve. A linearized equation was used to calculate the final saponin concentration [18]. The TSC (mg/g) was calculated based on the total saponin amount (T_{Sap}) and the initial leaf powder, using Eq. (1) [15]

$$TSC\ (mg/g) = \frac{mg\ T_{sap}}{g\ Jatropha\ sp.leaves\ extract\ powder} \quad (1)$$

2.5. Surfactant properties of the saponin extract from Jatropha curcas leaves

Saponin extracted from *Jatropha sp.* leaves was analyzed for its surfactant characteristics based upon the following analyses in comparison to pure saponin.

2.5.1. FT-IR analysis

A qualitative analysis of the extract was conducted by means of an FT-IR analyzer (Merk L160000A, USA, or Shimadzu) to identify the specific functional groups of saponins. The solutions of saponin extract were used as-is.

2.5.2. HPLC analysis

The presence of saponin in *Jatropha sp.* leaves extracts was measured using HPLC (LC20AD, Shimadzu, Japan) analysis, at the wavelength of 207nm. Here, the calibration curve of pure saponin was used as the standard [7].

2.5.3. Critical micelle concentration measurement of saponin from Jatropha curcas leaves

The critical micelle concentration (CMC) was determined based on the surface tension of the diluted saponin extracts (0.1-2 wt.%) using the capillary rise method [20]. CMC was identified as the lowest concentration where surface tension plateaued, indicating micelle formation. Pure saponin CMC served as control. In this method, when a liquid rises in a capillary tube, the surface tension (γ) can be calculated based on the capillary tube radius (r), liquid density (ρ), and capillary rise height (h) when $\theta \approx 0°$ ($\cos\theta = 1$) using Eq. (2-4) [21].

$$Upward\ force\ (F) = \pi . r^2 . h . \rho . g \quad (2)$$

$$Downward\ force\ (F) = 2\pi . r . \gamma . \cos\theta \quad (3)$$

$$\gamma = \frac{r . h . \rho . g}{2} \quad (4)$$

2.6. Micellar solubilization of dyes

Dye solubilization was assessed based on the method by Samal et al. [18] using remazol red RB and remazol blue TQ as the model of reactive dyes. Saponin extract at optimal yield and pure saponin were used as surfactants. Solutions were prepared at 1000–16,000 ppm, covering concentrations below, at, and

above the CMC. Each was mixed with 10,000 ppm dye and shaken at 200 rpm for 24 h at 28 ± 2°C. After incubation, samples were centrifuged (DM0412, DLAB Scientific Inc.), and the supernatants were analyzed for dye and saponin concentrations via UV-Vis spectrophotometry. Absorbance was measured at each λ_{max} of the dyes and quantified using calibration curves. The measured dye concentrations, considered as the solubilized dye, were used to calculate solubilization capacity using Eq. (5) where S_{dye} and S_{CMC} are the dye concentration at saponin concentration and CMC.

$$SP = \frac{S_{dye} - S_{CMC}}{C_{saponin} - CMC} \quad (5)$$

2.7. Micellar solubilization measurements

The value of molar solubilization power (SP) can explain the dye solubilization properties of the saponin micelles, representing the amount of solubilized dye per moles of micellized saponin. SP was calculated from the slope of the linear plot between solubilized dye concentration and saponin concentration. It may also be expressed in mass units (g/g), after conversion using the molecular weights of both dye and surfactant. To assess solubilization effectiveness, the partition coefficient (*Km*) was also determined, expressing the ratio of dye distribution between micellar and aqueous phases [18], calculated using Eq. (6):

$$Km = \frac{SP}{[Scmc].Vw.(1 + SP)} \quad (6)$$

where Vw is the molar volume of water ($1{,}805 \times 10^{-2}$ L.mol^{-1} at 298.15K) and S_{CMC} is dye concentration at CMC. The feasibility of the dye solubilization on surfactant micelles was presented by Gibbs free energy (ΔG), calculated with following eq. (7).

$$\Delta G = -R.T.\ln Km \quad (7)$$

3. Results and Discussion

3.1. FTIR analysis of saponin

The FTIR spectra of *Jatropha* sp. leaf powder, leaf extract, and pure saponin were analyzed and compared with data from literature [14,18,22]. Fig. 1 presents the IR spectra for the samples. The FTIR spectrum of the saponin standard showed absorption bands at 3393.9 cm^{-1} for hydroxyl (O-H) stretching, 2932.16 cm^{-1} for carbon-hydrogen (C-H) stretching, 1609.73 cm^{-1} for carbon-carbon double bond (C=C) stretching, and 1077.05 cm^{-1} for carbon-oxygen (C-O-C) stretching. Similarly, all samples exhibited spectra comparable to the saponin standard with O-H stretching ranging from 3307.13 cm^{-1} to 3292 cm^{-1}, C-H stretching at 2919.56 cm^{-1}, C=C stretching from 1635.69 cm^{-1} to 1624.84 cm^{-1}, and C-O-C stretching between 1038.82 cm^{-1} and 1016.62 cm^{-1}. The strong similarity between the FTIR spectra of the saponin standard and *Jatropha* sp. leaf powder and extract confirmed the presence of saponins in the samples.

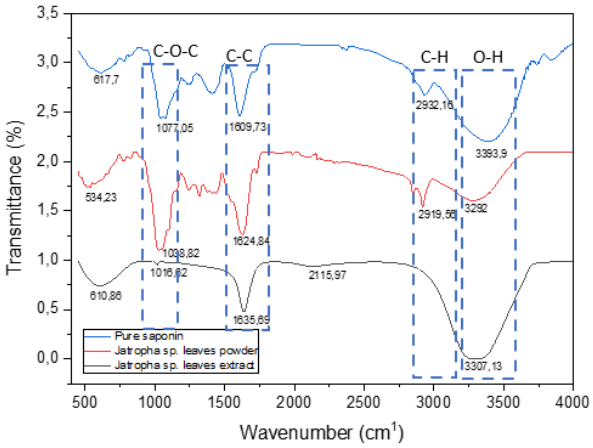

Fig. 1. FTIR transmittance spectra of pure saponin, *Jatropha sp.* leaves powder and extract

3.2. HPLC analysis

Further confirmation of saponin presence was obtained through HPLC analysis. Saponin extract was analyzed and compared with the database of pure saponin chromatogram. Figure 2 shows the HPLC chromatogram of *Jatropha* sp. leaf extract. HPLC analysis confirmed the presence of saponins in *Jatropha* sp. leaf extract, as indicated by two main peaks at retention times of approximately 1.331 and 2.000 minutes, which closely matched the retention times of the saponin standard (1.179 and 1.990 minutes). These findings are consistent with the previous study, which evaluated saponin-based natural surfactants for enhanced oil recovery [23]. The study reported a saponin standard retention time of 2.1 minutes, with shorter retention times potentially attributed to differences in extraction techniques. Additionally, the third peak and its shift suggested the presence of other compounds, indicating that it no longer corresponded to saponins in *Jatropha* sp. The HPLC results as presented in Figure 2 further confirmed the presence of saponins in the extract.

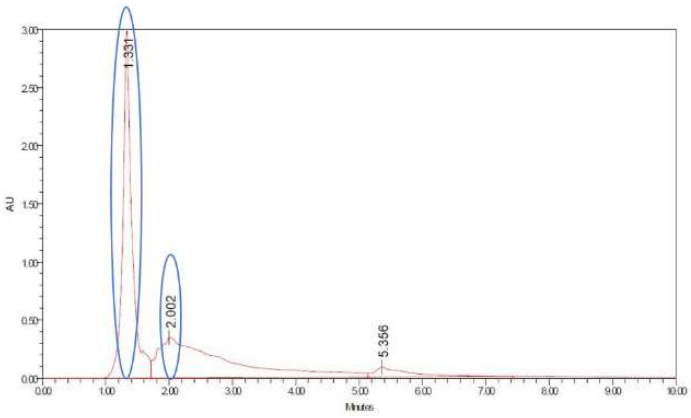

Fig. 2. HPLC chromatograms of *Jatropha sp.* leaves extract 4wt%

3.3. Microwave-assisted extraction of saponin from Jatropha sp. leaves

The extraction process was carried out via the MAE method with water as the solvent chosen due to its cost effectiveness, high dielectric constant, and environmentally

friendly nature [2,22,24]. The results of the extraction process for various extraction conditions are shown in Fig. 3, 4 and 5.

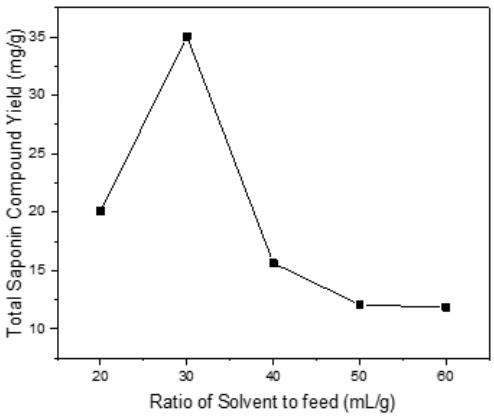

Fig. 3. Effect of the solvent-to-feed ratio on the TSC of *Jatropha sp.* leaves extracts at 363.15 K for 3 min

Fig. 3 shows the effect of solvent-to-feed ratio on the result of the extraction. The solvent-to-feed (S:F) ratio is a crucial parameter in MAE [25]. Proper S:F ratio ensures adequate wetting of the plant matrix and efficient microwave absorption [13]. The TSC increased from 20.06 mg/g to 35.04 mg/g as the S:F ratio increased from 20:1 to 30:1 (mL/g), showing that the increase of S:F ratio improved both wettability and microwave absorption within the material matrix. However, further increase on the ratio resulted in excessive water content, which absorbed more microwave energy due to its dielectric properties, ultimately reducing the total yield of saponin compounds as shown by the decrease of the TSC to 11.84 mg/g at higher S:F ratios of 60:1. Additionally, an increased solvent volume generate more heat within the microwave system, potentially causing the evaporation of thermolabile compounds [16]. Thermolabile compounds degrade when continuously exposed to high temperatures at their boiling point [26]. The previous study confirmed that combining an optimized solvent system with MAE is an effective strategy for bioactive compound recovery [27]. Similarly, another study demonstrated that saponin content is significantly determined by extraction time, solvent concentration, irradiation time, and solvent-to-sample ratio [28].

Irradiation time is another critical factor in MAE. While increasing irradiation time generally enhances yield, exceeding an optimal threshold can lead to reduced extraction efficiency. Figure 4 presents the TSC of *Jatropha* sp. leaf extracts at various irradiation times. The highest TSC of 35.04 mg/g was obtained at 3 minutes of irradiation. However, yields declined at 6 and 9 minutes, likely due to the degradation of bioactive saponins. These findings align with previous studies indicating that while MAE yield initially increased with irradiation time, prolonged exposure at high temperatures (363.15 K) could cause the significant degradation of saponins and other biologically active compounds [29,30].

Extraction temperature plays a vital role in determining yield. Higher temperatures commonly accelerate extraction, reducing the required processing time. However, in some cases, excessive temperatures can degrade sensitive components. The effect of temperature on TSC is shown in Fig. 5. Initially, TSC increased with temperature; nevertheless, at the extraction temperature of 373.15 K, TSC decreased to 20.02 mg/g, indicating thermal degradation. These findings demonstrated a general trend in extraction efficiency where yield improves up to an optimal temperature before declining due to compound degradation.

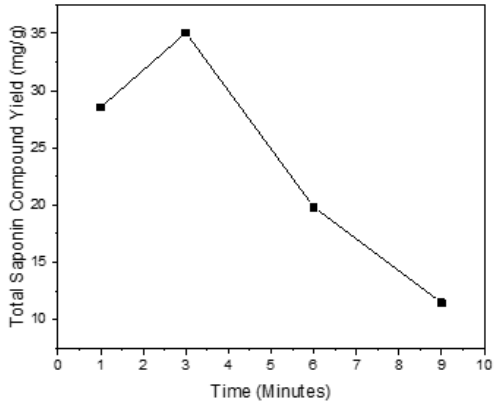

Fig. 4. Effect of irradiation time on the TSC of the *Jatropha sp.* leaves extracts at a temperature of 363.15 K and the solvent-to-feed ratio of 30 mL/g

Moreover, the saponin solubility increases along with the increasing temperature, as higher temperatures enhance solvent penetration, allowing saponins to dissolve more readily and migrate from the plant matrix into the bulk phase. Additionally, elevated temperatures increase the porosity of the solid matrix and reduce solute viscosity, making it easier for saponin molecules to interact with the solvent, and be extracted [31]. However, the excessive rise of the temperatures decreases the calculated TSC, indicating the decomposition of saponin compounds [30].

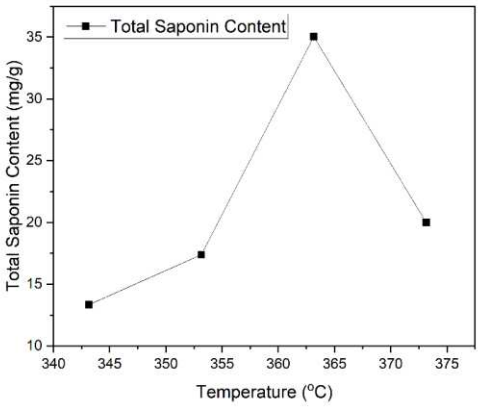

Fig. 5. Effect of extraction temperature on total saponin compound yield

The TSC was analyzed spectrophotometrically, which relied on the chromophoric groups of the saponin e.g. triterpenoid/steroid core or conjugated double bonds [32]. Intact saponins strongly absorb light at their characteristic wavelength. However, if degradation disrupts the conjugation of the molecule, this will promote cleavage on the glycosidic bonds, and produce smaller molecules that absorbs less light, thus reducing the detected concentration [33]. Consequently, this reduction reflects compound alteration. Similar result was also reported in phenolic extraction, where temperature initially

gave a positive shift on the extraction result and showed a decrease after the optimum temperature condition achieved [34]. The molecular structure of saponin is depicted in Fig. 6.

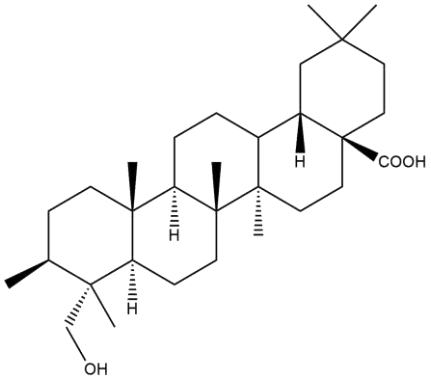

Fig. 6. Molecular structure of saponin

3.4. Analysis of CMC of saponin and micelle property

In a liquid solution, surfactant monomers aggregated into micelles at a specific concentration are known as the critical micelle concentration (CMC). These micelles play a crucial role in adsorbing organic solutes, such as dyes [35]. In this study, the CMC value was determined by analyzing the decline in the water surface tension by varying the saponin concentrations, as presented in Figure 7.

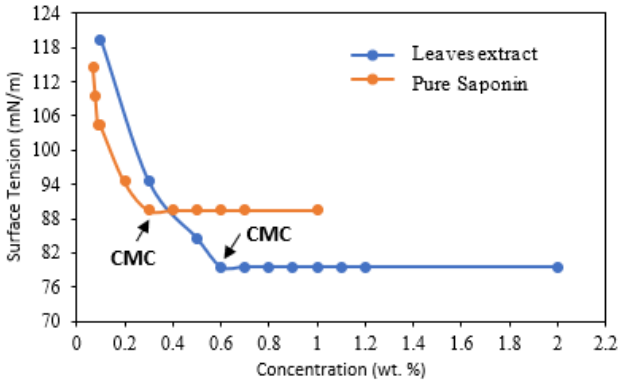

Fig. 7. Measurement of CMC of surfactants by capillary rise method

Generally, water surface tension decreases with the addition of saponin into solutions. However, beyond a certain concentration, surface tension remains nearly constant despite the continued addition of saponin, indicating the critical micelle concentration (CMC). Pure saponin showed CMC of 0.3 wt.%, aligning with the reported CMC range provided by the manufacturer at 0.001–0.1 wt.%. Saponin extracted from Jatropha sp. Leaves obtained higher CMC of 0.6 wt.%. This value was calculated based on the total saponin yield obtained under optimal extraction conditions. The higher CMC of the extract compared to pure saponin may be attributed to the presence of impurities in the extracted solution. Similar findings have been reported in previous studies where extract solutions exhibited higher CMC values than that of pure saponin [8,36]. Assuming that the molecular weight of the extracted and pure saponin was identical, the CMC values could be expressed in millimolar (mM) with 4.86 mM for extract saponin and 2.45 mM for pure saponin. The micelle formation mechanism is illustrated in Fig. 8.

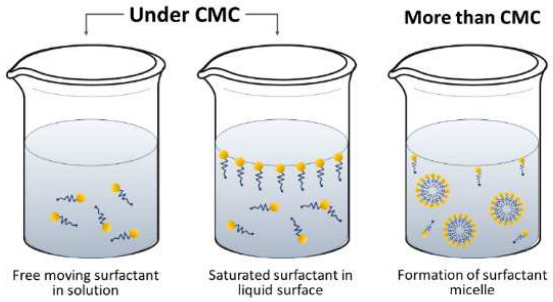

Fig. 8. The illustration of the micelle form mechanism

3.5. Solubilization of dyes

A dye solubilization experiment was conducted using saponin extracted from Jatropha sp. leaves as a natural surfactant to assess its micelle-forming capabilities. The effect of saponin concentration on the solubility of Remazol Red RB and Remazol Blue TQ is illustrated in Fig. 9.

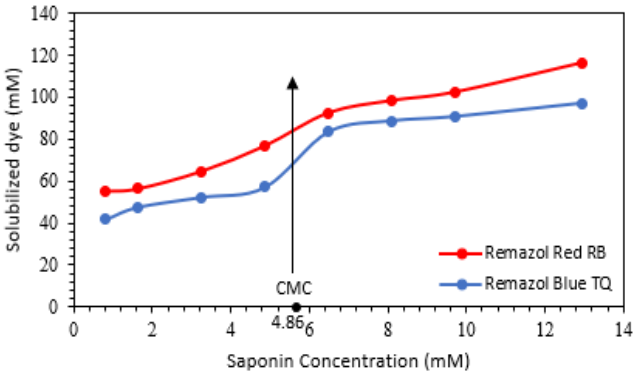

Fig. 9. Effect of saponin concentration (in mM) on the solubility of remazol red RB and remazol blue TQ at 28 ± 2°C

The reactive dyes solubilization capacity of the saponin micelles was analyzed at concentrations below and above the CMC. The previous report found that a solubilization period of 18–24 hours was sufficient to achieve equilibrium as no significant increase in dye solubilization was observed beyond 24 hours [18,37]. Whereas, this study confirmed that equilibrium reached within 24 hours.

Fig. 10. depicts the solubilized dye (in mM) as the function of TSC. The results demonstrated the increase of dye solubility by the addition of surfactant concentration, even beyond the CMC, indicating the significant role of surfactant micelles in dye solubilization. Micelle agglomerates may also adsorb dye molecules and this furthermore can increase solubilization [38]. These findings align with a previous study, reporting that the micelles solubilized dyes by facilitating the solute movement into their structures [39]. This process involved the interactions of surfactant, water and dyes. Surfactant forms amphiphilic micelles, which bind solutes at concentrations above the CMC. Micelles facilitate the dissolution of highly water-soluble

solutes. According to Huang et al. [40] surfactant micelles consist of three distinct layers: hydrophobic inner core, CH_2-based palisade layer and hydrophilic outer layer. However, the exact location of solubilization within non-ionic surfactant micelles remains unclear. Some researchers have proposed that solubilization occur in both hydrophilic-hydrophobic regions [38]. While, the relative solubilization in these regions depends on the polarity of the dyes [41]. The polar outer portion leading to the preferential retention of polar solutes. Consequently, polar molecules, such as dyes, may be partially incorporated into the outer region of the surfactant micelles [38].

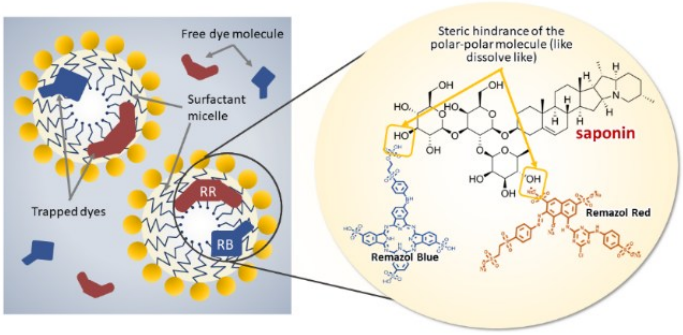

Fig. 10. The mechanism of dye solubilization of saponin for remazol red RB and remazol blue TQ

Furthermore, the molar solubilization power (SP) of saponin was 5.40- and 5.08-mM dye/mM saponin for Remazol Red RB and Blue TQ, respectively. The higher solubilization capacity of saponin micelles for Remazol Red RB compared to Remazol Blue TQ attributed to the molecular weights differences between the dyes. The structural difference between the dyes also influenced their solubilization behavior. Since the molecular structure of Remazol Red RB was smaller than that of Remazol Blue TQ, it was expected to exhibit higher solubilization efficiency.

Table 1. Molar solubilization power (SP) of saponin for remazol red RB and remazol blue TQ, ln Km, and Gibbs free energy of solubilization (DG) at 28 ± 2°C

Type of Reactive Dyes	*Solubilization Power* (SP) (mM/mM)	ln Km	*Gibbs Free Energy* (ΔG) (kJ mol^{-1})
Remazol red RB	5.40	9.00	-22.32
Remazol blue TQ	5.08	9.19	-22.77

At last, understanding thermodynamic parameters, such as Gibbs free energy (ΔG), is essential for evaluating the solubilization feasibility. The calculated solubilization Gibbs free energy (ΔG) was -22.32 and -22.77 kJ/mol for Remazol Red RB and Blue TQ, respectively. The negative values indicated the thermodynamically feasible solubilization of both dyes occurred spontaneously. Table 1 summarizes the SP, ΔG, and ln Km values for both dyes. While, the dye solubilization mechanism is depicted in Fig. 10.

4. Conclusion

This study demonstrated the potential of *Jatropha curcas* leaf extract as a sustainable surfactant and solubilizing agent for reactive dyes. Microwave-assisted extraction significantly enhanced the yield of saponins with the highest yield of 35.04 mg/g. FTIR analysis confirmed that the functional groups of the extracted saponin were comparable to those of pure saponin. The CMC of the extracted saponin (0.3 wt.%) was lower than that of pure saponin (0.6 wt.%), indicating superior surfactant properties. Meanwhile, thermodynamic analysis revealed negative ΔG for dye solubilization, confirming feasible and spontaneous dye solubilization. The results of the study highlights the potential of *Jatropha curcas* leaf extract as an eco-friendly alternative to synthetic surfactants, particularly in applications such as reactive dye solubilization.

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