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Research Paper

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Synthesis and evaluation of the surface and thermodynamic properties of

nonionic surfactants obtained from vegetable oil waste

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Abstract : Nonionic surfactants were produced from fatty acids in vegetable oil refining waste. By utilizing the fatty acids obtained through the process of refining vegetable oil as starting materials for surfactant synthesis and using PEG (600 and 1500) as co-surfactants provides an environmentally friendly and sustainable method for the production of nonionic surfactants. Research shows the potential of using waste to produce surfactants to contribute to the development of the circular economy. The final nonionic surfactants were characterized using infrared spectroscopy and 1HNMR spectroscopy. The results showed that synthetic surfactants exhibit good surface properties. Various analytical methods will be employed to assess the surface and thermodynamic characteristics of the synthesized surfactants Analytical techniques like surface tension measurement and surface determination have been utilized to evaluate the properties of the surfactants. The collected data has been analyzed to gain insights into the effectiveness and performance of the surfactants in diverse applications. (eg, emulsification, foaming), making it suitable for a variety of industries and businesses.

KEYWORDS: Fatty acid; Nonionic surfactany; vegetable oils waste

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I. INTRODUCTION

Non-ionic surfactants(Cortés et al. 2021) which are widely used in industries such as personal care (Guin et al. 2022), household products(Munir et al. 2014), and agrochemicals(Fouquet et al. 2017) are valued for their surface properties like emulsification, wetting, and dispersion (Rosen M. J. 2004). They have become essential in applications like detergents, cosmetics, and pharmaceuticals due to their versatility and effectiveness in emulsification, dispersion, dissolution, and stabilization (G Hassabo 2018). Non-ionic surfactants offer versatility and find applications across various functions, such as emulsification, dispersion, solubilization, and stabilization. (Adewuyi et al. 2012). In recent years, there has been a notable rise in the popularity and widespread adoption of non-ionic surfactants (Munir et al. 2014) due to their effectiveness, low toxicity, and biodegradability(Yara-Varón et al. 2017b). Non-ionic surfactants are preferred over anionic and cationic surfactants because they are mild, low foaming, and compatible with other ingredients (Yelubay et al. 2020). These surfactants are often produced using fatty acids derived from vegetable oil refining processes(Brica, Klavins, and Zicmanis 2016). Vegetable oil refineries generate significant amounts of fatty acids as by-products (Shu et al. 2010), These raw materials can be effectively utilized for the production of non-ionic surfactants. (Yara-Varón et al. 2017a). The utilization of fatty acids obtained from waste generated during the refining of vegetable oil to produce non-ionic surfactants presents a sustainable and eco-friendly solution. This method reduces dependency on non-renewable resources and offers a valuable use for waste materials, thereby enhancing their value and utilization (Chemat et al. 2019). Non-ionic surfactants based on fatty acids(Megahed et al. 2010) provide a sustainable and efficient alternative to petroleum-derived surfactants (Johansson and Svensson 2001). By adjusting the composition of fatty acids, it is possible to customize the properties of these surfactants (Yea et al. 2021), degree of unsaturation, and molecular weight (Gomna et al. 2019). By utilizing

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fatty acids from renewable sources like vegetable oil refinery waste (Biermann et al. 2021), the development of non-ionic surfactants with improved performance and reduced environmental impact is possible (El-dougdoug, Azab, and Torky 2019). Using fatty acid refinery waste from vegetable oil (Kumar et al. 2012) not only reduces waste but also conserves resources and minimizes greenhouse gas emissions associated with the production of petroleum-based surfactants (Philosophy 2020) Vegetable oil refineries generate substantial amounts of fatty acids as by-products (Piloto-Rodríguez et al. 2014). These fatty acids can be efficiently utilized as raw materials for the.

II. MATERIALS AND METHODS

2.1. Materials

Fatty acids ((Myristic acid, palmitic acid, stearic acid, oleic acid and linoleic acid), were purchased from Al-Gomhuria co. Egypt. Polyethylene glycol (600-1500) purchased from Merck India. p-Toluene sulfonic acid was procured from Sigma-Aldrich Chemicals Co. High-grade solvents including petroleum ether (40-60°C), n-hexane, and methanol were obtained from Algomhoria Chemicals Co.

2.2.Synthesis of esterified fatty acid nonionic surfactants

The synthesis of esterified fatty acid nonionic surfactants from vegetable oil waste was carried out through the following series of steps.

2.3. Synthesis of fatty acids monoester.

A mixture consisting of 0.1 mol of fatty acids (such as linoleic acid, oleic acid, palmitic acid, and stearic acid) and 0.1 mol of polyethylene glycol (600 and 1500) was subjected to refluxing using dry xylene as a solvent and 0.01% p-toluene sulphonic acid as a catalyst. The reaction was performed under Dean-Stark trap conditions until the anticipated amount of water (0.1 mol) was separated. Subsequently, the resulting mixture was neutralized using sodium carbonate and subsequently washed twice with distilled water. Afterward, the mixture was dried with anhydrous sodium sulphate, and the solvent was removed through distillation (Aiad et al. 2016). The chemical structures of the synthesized monoesters were verified using Fourier-transform infrared spectroscopy (FTIR). (El-Dougdoug, Azab, and Torky 2019a).

2.4. Surface properties of Nonionic

2.4.1. Surface Tension

The surface tension (γ) of freshly prepared solutions of various concentrations of nonionic surfactants was determined using a De Nooy ring tensiometer (Kruss-K9). The surface tension of distilled water, which was utilized prior to preparing the surfactant solution, was measured. Platinum rings were submerged in water, cleansed with acetone, and subsequently subjected to brief heating to eliminate any residual acetone. (Shaban et al. 2015). The average value of three consecutive measurements was recorded for each reading

2.4.2Measurements of Foaming Properties (Mahapsari 2013)

In both application and research laboratories, this test is extensively employed to assess the foaming stability of surfactant solutions and to determine the presence or absence of antifoaming agents. In our specific experiment, we employed this test for the aforementioned purposes (Abd El-Ghaffar, Sherif, and Taher El-Habab 2017), An amount of the prepared nonionic surfactant, 0.1% by weight of the aqueous surfactant solution, was preemulsified and stirred for 10 minutes using a magnetic stirrer. The resulting emulsion was further homogenized by hand holding several times before being transferred to the glass cylinder and circulated for 20 seconds. The degree of foaming in the solution was assessed by measuring the initial volume of bubbles after discontinuing the circulation of the aqueous surfactant solution. Dynamic foam is generated in a circular shape and attains a certain height or volume within approximately 30 seconds of circulation. The height of the dynamic foam at a stable state is referred to as the Dynamic Foam Height (DFH) and is utilized as a measure of the solution's foaming capacity. Standard methods are employed to determine foam stability (Saito, Sato, and Anazawa 1989): Foam stability (%) = (Foam volume after 5 mins/ Foam volume after 0 mins) *(100)

Foam volume was measured in milliliters and foam stability was evaluated by measuring time at 28 $^\circ$ C (Ross and Miles, n.d.).

2.4.3. Stability to hydrolysis

10 mL of surfactant solutions (containing 10 mmole) were mixed with either 10 mL of 0.05 N sodium hydroxide or 10 mL of 2 N sulfuric acid in phenol tubes. The phenol tubes were subsequently placed inside a temperature-controlled system set at 40 °C. The hydrolysis resistance of the surfactant was evaluated by measuring the time the sample solution became cloudy (El-Dougdoug, Azab, and Torky 2019b).

<u>2.4.4.</u> Determination of the Cloud Point of the Prepared Nonionic Surfactants</u> (El-Shattory, El-Mawla, and El-Hamide 2018)

Cloud point is a common parameter used to describe nonionic surfactants. It is determined by heating a 1% by weight solution of surfactant in deionized water until cloudy and then recording the temperature at which the solution becomes clear upon cooling. Repeat this process at least three times to determine the cloud point with an accuracy of ± 0.2 °C. The reported cloud point value is the average of 3 consecutive measurements called the apparent cloud point (C.P.). (Schott 2001).

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2.4.5. Emulsifying Capability

To examine the ability of emulsification, we utilized 5 mL volumes of a 2% aqueous surfactant solution. The testing period spanned from 0 to 24 hours. To determine the separation rate, we employed the following formula.:

R_s =Vs * 100/V₀

where V_0 is the initial volume in mL of emulsion, R_s % is the separation rate percent, Vs is the volume of water in mL separated from the emulsion.

2.4.6. Hydrophilic-Lipophilic Balance (HLB) of Prepared

No<u>nionic Surfactants</u>

To relate the structure and surface activity of surfactant molecules, Griffin developed the concept of hydrophilic-lipophilic balance (HLB).(Zaimee et al. 2020). For non-ionic surfactants, HLB values can be calculated or determined empirically. The experimental procedure is lengthy and arduous and was described several years ago. (Abd El-Ghaffar et al. 2017). HLB values were calculated with the Griffin formula based on the ethylene oxide moiety in the molecule as follows

$$HLB = 20X \frac{MW_H}{MW_H + MW_I}$$

where $MW_H = Mol. W_t$ of the hydrophilic part and $MW_L = Mol W_t$ of lipophilic part. Table 3 shows the HLB values calculated with the Griffin formula for the novel prepared surfactants. It is clear from Table 3 that HLB generally increases with increasing number of ethylene

oxide units per surfactant molecule (Rosen, 2004)

2.5. Gas-liquid chromatography (GLC)

The mixed fatty acid methyl ester samples were subjected to gas chromatography (GLC) analysis using a Perkin Elmer Auto System XL equipped with a flame ionization detector (FID). A fused silica capillary column (ZB-5) with dimensions of 60 m \times 0.32 mm I.D. was employed. The initial oven temperature was set at 150 °C, followed by a programmed temperature increase from 150 to 240 °C at a rate of 30 °C/min. Helium gas was used as the carrier with a flow rate of 1 mL/min. The injector and detector temperatures were set to 230 °C and 250 °C, respectively. The split ratio was 1:10, and a sample size of 2 μL was utilized. The identification of GLC peaks was performed by comparing the obtained chromatograms with those of standard fatty acid methyl esters from Sigma, USA, as well as flavor and odor lab from the National Research Centre in Dokki, Cairo, Egypt 2.6The structural confirmation of the synthesized compounds

2.6.1.FTIR Spectra The FT-IR spectra of the synthesized compounds were obtained in both liquid and solid states using a Thermo Nicolet iS10 FT-IR spectrophotometer. The measurements were conducted on a KBr disk at (the Faculty of Science, Benha University, Benha, Egypt).

2.6.2.¹H NMR spectra

The 1H NMR spectra were acquired using a Bruker Avance (III) 400 MHz NMR spectrometer from Switzerland. The spectra were recorded at 298 K with a total of 128 scans. Deuterated dimethyl sulfoxide (DMSO-d6) and/or deuterated chloroform (CDCl3-d) were used as solvents, and tetramethylsilane (TMS) was utilized as an internal reference compound



III. RESULTS AND DISCUSSION

3.1. Determination of Fatty acids composition

The fatty acid composition was determined through Gas-Liquid Chromatography (GLC), as depicted in Figure 1. The resulting data has been collected and organized in Table 1. The analysis reveals that the majority of the fatty acids consist of approximately 85.66% unsaturated fatty acids, with a smaller proportion comprising saturated fatty acids. The chemical measurements conducted confirmed the analysis, as the oil exhibited a high iodine value. This high iodine value indicates a significant proportion of unsaturated fatty acids in the oil. Five nonionic surfactant compounds were synthesized using different fatty acids, namely, meristic acid, palmitic acid, stearic acid, oleic acid, and linoleic acid. These fatty acids were derived from vegetable oil waste



Fig. 1 GIC					
Fatty Acids	Percent				
C14:0	1.45%				
C16:0	12.89 %				
C18:0	02.86 %				
C18:1	25.23 %				
C18:2	57.57 %				

Table.1 Composition of fatty acid mixture

3.2. Characterization: -

3.2.1. The FTIR spectra of the mono oleate ester (PEG 600).

The FT-IR spectra verify the presence of the anticipated functional groups in the synthesized ester, as depicted in Figure 2. These functional groups are indicated by the characteristic bands observed at 2924 cm-1 and 2857 cm-1 (vC-H aliphatic fatty chain), 1736 cm-1 (vC=O of ester), 1648 cm-1 (vC=C stretch aliphatic fatty chain), and 1111 cm-1 (vC-O-C stretching).



Fig.(2)The FTIR spectra of "Ethoxylated mono Oleate 600 3.2.2. The FTIR spectra of the mono oleate ester (PEG 1500).

The expected functional groups in the synthesized ester were confirmed by the FT-IR spectra, as illustrated in Figure 3. The spectra exhibited characteristic bands at 2926 cm-1 and 2854 cm-1 (vC-H aliphatic fatty chain), 1738 cm-1 (vC=O of ester), 1645 cm-1 (vC=C stretch aliphatic fatty chain), and 1112 cm-1 (vC-O-C stretching).



Fig.3 The FTIR spectra of "Ethoxylated mono Oleate 1500

3.2.3. ¹H NMR spectra

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The data obtained from the 1H-NMR spectra validate the anticipated distribution of hydrogen protons in the synthesized compound δ (ppm): 0.87 (t, 3H, CH3), 1.27 (m, 28H, (CH2)14CH3), 1.62 (m, 2H, COOCH2CH2(CH2)14), 2.33 (t, 2H, CH2COO- CH2(CH2)14), 3.25 (t, 2H, COOCH2CH2OCH2), 3.64 (t, 2H, COOCH2CH2OCH2), 4.24 (t, 2H, COOCH2CH2OCH2), 5.35 (s, 2H, NH2) (fig.4)



Fig.4¹H NMR spectra of Ethoxylated mono oleate (PEG 600)

3.<u>3. Properties</u>

3.3.1.Surface tension:

As the hydrophobicity of a surfactant increases, its solubility in water decreases, resulting in an increase in the surface tension of the system. (Negm et al. 2013) Tab2.

3.3.2. Critical Micelle Concentration (CMC):

Table 2 provides the estimated critical micelle concentration (CMC) values for various nonionic surfactant molecules. Additionally, a graph is included to depict the relationship between the surface tension (γ) of aqueous surfactant solutions and the natural logarithm of their concentration (-log[conc]) measured in moles per liter. was in Fig 5

3.3.3. Effectiveness (π_{CMC}) and Efficiency (P_{C20})

The effectiveness of the synthesized nonionic surfactants can be evaluated by determining the difference between the surface tension of distilled water (γ_0) and the surface tension of the surfactant solution at its critical micelle concentration (γ CMC), as presented in Table 2. This estimation can be carried out using the following equation.

$$\pi_{CMC} = \gamma_o - \gamma_{CMC}$$

3.3.4. Surface pressure (π_{cmc}) :

The surface pressure serves as a suitable parameter for assessing the efficacy of surfactants by measuring their ability to reduce the surface tension of water. The calculation of the critical micelle concentration (π cmc) in millinewtons per meter (mN/m) can be determined using the following equation:

$$\pi_{\rm cmc} = \gamma_{\rm o} - \gamma_{\rm cmc}$$

Where γ_0 is the distilled water surface tension and γ_{cmc} is the surfactant solution surface tension at CMC. The ideal synthesized surfactant is the one that demonstrates the greatest reduction in surface tension at the critical micelle concentration (CMC). (Shaban et al. 2014)

3.3.5. Maximum surface excess (Γ_{max}) and minimum surface area (A_{min}) Gibb's equation can be used to calculate the maximum surface excess, denoted as Γ max, of a synthesized nonionic surfactant. This value represents the accumulation of surfactant molecules at the interface between air and water. (Takeshita, Shimohara, and Maeda 1982)

$\Gamma_{\text{max}} = 1/2.303 \text{ nRT} (\partial \gamma / \partial \log C)$

Where, R = gas constant (8.314 J mol-1 k - 1) and T = t+273 (°K); The value of "n" represents the number of ionic species that are concentrated at the interface and changes with the surfactant concentration in the solution. In Table 2, the Fmax values of the synthesized nonionic surfactant were calculated at various temperatures. The

average area occupied by each adsorbed molecule of a surfactant can be determined using the following equation. (Baddini et al. 2007). $A_{min} = 10^{14}/N_A \Gamma_{max}$

Where N is Avogadro's number. By inspecting the data in Table (2) As the temperature of the measurements rises, the amount of surface area per molecule at the interface between the aqueous solution and air also increases. This implies that higher temperatures lead to a decrease in the number of molecules present at the interface. Moreover, the surface area values at the interface for the synthesized nonionic surfactant molecules exhibit a gradual increase.

3.3.6. Foaming properties:

Table (3) contains the recorded foaming volume of the prepared nonionic surfactant. The synthesized nonionic surfactant demonstrates favorable foaming characteristics in comparison.

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3.3.7 Stability to hydrolysis

Table (3) displays the results of exposing the synthesized nonionic surfactant to acidic and basic environments in order to investigate its hydrolysis. The synthesized surfactant demonstrated decreased stability in an acidic medium. However, all the surfactants studied showed strong stability in a basic medium. (Weil et al. 1963).

property Surfactant	γ mN/m	π _{смс} mN/m	$\Gamma_{max} \times 10^{-4}$ mol /cm ²	A _{min} ×10 ⁻⁷ (nm ²)	СМС	ΔG_{mic}
Fatty ethoxylated stearate (M.wt.1500)	41	31.86	7.82328	2.122	0.025	-20.69
Fatty ethoxylated oleate (M.wt.1500)	37.5	35.36	8.4378	1.967	0.0125	-24.58
ethoxylated fatty acid mixture (M.wt.1500)	40	32.86	5.92127	2.803	0.00625	-28.47

Table 2: The surface characteristics of the surfactants that were produced synthetically.

Surfactant	Foamcloud pointheight(°C) 1 wt.%(mm)1wt%	Foam stability (mm) 1.0 wt.%			Emulsion stability	Stability to hydrolysis (hr:min)		HLB	
		(mm) 1wt%	0 min	3 min	5 min	(hr: min) 1 wt.%	Acidic (2.0 N)	Alkaline (0.05 N)	
Fatty Ethoxylated Stearate (M.wt.1500)	>90	250	250	190	170	00:55	26:10 hr.	61:20 hr.	16.9
Fatty EthoxylatedOleate (M.wt.1500)	>90	240	240	140	130	01:30	25:.09 hr.	55:54 hr.	17

 Table 3: The surface properties of the synthetically produced surfactants were investigated

 (5): urface tension was investigated in relation to the logarithm of the concentration for the synthetic surfactant





Fig (6): urface tension was investigated in relation to the logarithm of the concentration for the synthetic surfactant. (oleate 1500)



Fig (7 urface tension was investigated in relation to the logarithm of the concentration for the synthetic surfactant (Mono fatty acid mixture)

V-CONCLUION

To sum up, the use of vegetable oil wastes to produce nonionic surfactants has become increasingly popular because of their sustainable and environmentally friendly characteristics. By utilizing these oil wastes, the production of nonionic surfactants can help reduce the negative environmental effects typically associated with petroleum-based surfactants. The synthesized nonionic surfactants have demonstrated exceptional surface-active properties, making them suitable for a wide range of applications such as detergent formulations, emulsifiers, and dispersants.

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