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Sustainable recovery of crude fatty acids from cotton soapstock waste through ultrasonic saponification: Rheological and FTIR assessment

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ABSTRACT

Cotton soapstock, a by-product of cottonseed oil alkali refining, constitutes an environmental burden while simultaneously representing a valuable source of crude fatty acids (CFA). Conventional alkaline saponification requires substantial energy inputs and exhibits incomplete triglyceride hydrolysis. The present study evaluated ultrasonic process intensification (40 kHz) as an alternative approach for CFA recovery. Soapstock obtained from the "Istiqlol" cotton oil plant (Urgench, Uzbekistan) was saponified under both classical and ultrasonic conditions at temperatures of 65–95 °C and NaOH stoichiometric ratios of 1× to 4×. Saponification degree was determined by ISO 3657:2002, dynamic viscosity by ISO 2555:2018 (Anton Paar MCR 302 rheometer), density at 20 °C by pycnometry (GOST 18995.1-73), and foaming coefficient by standard protocol. FTIR spectra were acquired in ATR mode (Shimadzu IRTracer-100). Ultrasonication at 85 °C with 2× NaOH achieved a saponification degree of 97.2 ± 0.8%, equivalent to classical saponification at 95 °C, representing an energy saving of 8–12%. Dynamic viscosity decreased from 1.85 to 1.01 ± 0.04 Pa·s under optimal ultrasonic conditions due to acoustic cavitation-induced emulsification. The foaming coefficient was reduced by 56.3% relative to the classical method. FTIR analysis confirmed complete saponification: the ester C=O band at 1735.62 cm^{-1} disappeared and a strong COO^- stretch emerged at 1603.52 cm^{-1}. Strong inverse correlations were established among process parameters ($r = -0.91$ to -0.94, $p < 0.001$), supporting the validity of the multi-parameter monitoring approach.

Keywords: cotton soapstock; alkaline saponification; ultrasound; fatty acids; rheology.

INTRODUCTION

Alkali refining of edible oils generates substantial quantities of soapstock as a by-product (Bhosle & Subramanian, 2005; Haas et al., 2003). Cottonseed oil refining produces approximately 50–120 kg of soapstock per tonne of refined oil, with the precise amount depending on the free fatty acid content of the crude oil and the efficiency of the refining process (Dijkstra, 2010; Vaisali et al., 2015; Gunstone, 2011). Cotton soapstock is a complex mixture comprising sodium salts of fatty acids, free fatty acids, residual triglycerides, phospholipids, moisture, and inorganic salts, with a fatty acid profile dominated by linoleic acid (~52%), palmitic acid (~24%), oleic acid (~18%), and stearic acid (~3%) (Haas, 2005; Suslick, 1990; Gogate & Pandit, 2004; Guillén & Cabo, 1997). This composition renders it a prime candidate for CFA production for application in soaps, animal feed supplements, lubricants, biodiesel, and oleochemical intermediates.

Conventional CFA recovery from soapstock involves alkaline saponification of residual

triglycerides followed by acidulation with mineral acids. Incomplete saponification yields acid oil of reduced quality and quantity. The classical process requires elevated temperatures (85–95 °C), extended reaction times (60–120 min), and considerable NaOH excess (up to 3× stoichiometric), resulting in high energy and reagent consumption (Chemat et al., 2011; Rohman & Che Man, 2011; Abismaïl et al., 1999; Deacon & Phillips, 1980; Vlachos et al., 2006).

Ultrasonic treatment represents a promising process intensification strategy based on acoustic cavitation: ultrasonic waves generate microbubbles that collapse violently, creating intense local conditions and microjets reaching velocities of 100–200 m/s, thereby substantially improving mass transfer in viscous emulsions. These effects accelerate saponification and enable equivalent conversion at lower temperatures (Vlachos et al., 2006; Cravotto & Cintas, 2006; Shamuratov et al., 2025; Shamuratov et al., 2026). Frequencies between 20 and 40 kHz are particularly effective for emulsion processing due to peak cavitation forces in this range. Notwithstanding increasing interest in ultrasonic lipid processing, comprehensive studies integrating rheological monitoring with FTIR structural analysis of soapstock saponification remain limited (Asilbekova et al., 1988; Lerma-García et al., 2009).

Uzbekistan is a major centre of cotton cultivation and oil refining. The "Urganch Cluster" enterprise ("Istiqlol" plant, Urgench, Khorezm) is among the large-scale facilities encountering challenges in soapstock waste management and fatty acid recovery.

The objectives of this study were: (1) to characterise the saponification of cotton soapstock under conventional and ultrasonic (40 kHz) conditions at 65–95 °C and NaOH ratios from 1× to 4×; (2) to evaluate four rheological parameters as process monitoring indicators; (3) to perform FTIR analysis of raw and saponified soapstock; and (4) to identify conditions for optimal, sustainable CFA recovery (Gogate et al., 2003; Guillén & Cabo, 1998; Chemat et al., 2004; Christy et al., 2019).

MATERIALS AND METHODS

Raw materials

Cotton soapstock was obtained from the alkaline refining section of the "Istiqlol" cotton oil plant (Urganch Cluster, Urgench, Khorezm Region, Uzbekistan). Samples were collected from three separate batches, combined, and stored in polypropylene containers at 4 ± 1 °C. All analyses were completed within two weeks of collection. Analytical-grade sodium hydroxide (≥98% purity) and deionised water (conductivity < 1 μS/cm) were used throughout.

Saponification procedure

Alkaline saponification experiments were conducted in a 500 mL jacketed glass reactor equipped with mechanical stirring (300 rpm) and thermostat-controlled temperature (± 0.5 °C). Classical saponification was performed at 65, 75, 85, and 95 °C with NaOH stoichiometric excess ratios of 1×, 1.5×, 2×, 2.5×, 3×, 3.5×, and 4×. Ultrasonic saponification was conducted by placing the reactor in an ultrasonic bath (40 kHz, energy density 35 W/L) (Chisti, 2003; Lozada-Ramírez et al., 2021; Mason & Peters, 2002) with all other parameters identical to the classical procedure. Reaction time was fixed at 60 min for parameter optimisation and 330 min for kinetic studies.

Rheological characterisation

Saponification degree was determined gravimetrically according to ISO 3657:2002 by back-titration of excess NaOH with standardised HCl (0.1 M). Dynamic viscosity was measured using an Anton Paar MCR 302 rotational rheometer (ISO 2555:2018; CP-25 geometry; shear rate γ = 10 s^{-1}; 70 °C) (Gibon et al., 2007; Dowd, 1996; FAO, 2023). Density was determined using a calibrated pycnometer (25 mL) at 20 ± 0.1 °C according to GOST 18995.1-73 (Yuldasheva et al., 2026; Rosen, 2004).

FTIR spectroscopic analysis

FTIR spectra were acquired using a Shimadzu IRTracer-100 in ATR mode (diamond crystal; 32 co-addition scans; 4 cm^{-1} resolution; 400–4000 cm^{-1}) with air as background reference. Analyses were performed on unprocessed soapstock and soapstock saponified under optimal ultrasonic conditions (85 °C, 2× NaOH) (Domb et al., 2024; Özgül-Yücel & Türkay, 2002; Boyjanov et al., 2026b; Buranova et al., 2025; Boyjanov et al., 2026a; Buranova et al., 2022).

Statistical analysis

Data are presented as mean ± standard deviation (SD; n = 3). One-way analysis of variance (ANOVA) with Tukey's Honest Significant Difference (HSD) post-hoc test was applied to assess statistically significant differences ($p < 0.05$). Pearson correlation coefficients were calculated. Statistical analyses were performed using IBM SPSS Statistics version 26.0 (Buranova, 2025; Boyjanov et al., 2026c; Mezger, 2014; Rao, 2010).

RESULTS AND DISCUSSION

Effect of NaOH stoichiometry and temperature on saponification degree

Three principal trends characterise saponification efficiency (Table 1, Figure 1). First, a marginal return effect was observed with increasing NaOH excess. From 1× to 2× NaOH, saponification improved substantially: under classical conditions at 85 °C, from 70.4% to 91.6% (+21.2 percentage points, p.p.); under ultrasonic treatment at 85 °C, from 79.3% to 97.2% (+17.9 p.p.). Beyond 2× NaOH, marginal gains were negligible — the classical method at 85 °C yielded only +1.7 p.p. from 2× to 4× NaOH. Tukey HSD testing confirmed that the difference between 2× and 2.5× was not statistically significant ($p = 0.18$).

At 2× stoichiometry, sufficient hydroxide ions are available to drive complete conversion, while excess alkali leads to diffusion limitations and pH-mediated inhibition effects.

Second, the advantage of ultrasonic treatment was most pronounced at lower temperatures. At 65 °C, ultrasonic conditions outperformed classical saponification by 10.6 p.p. (84.8% vs. 74.2%), whereas the difference narrowed to 2.7 p.p. at 95 °C. This temperature dependence is consistent with acoustic cavitation theory: at lower temperatures, diffusion-controlled saponification is

Table 1. Saponification degree (%, mean ± SD, n = 3) as a function of NaOH stoichiometric ratio and temperature under classical and ultrasonic (40 kHz) conditions

T (°C)	Method	1×	1.5×	2×	2.5×	3×	3.5×	4×
65	Classical	52.4±1.8	61.8±1.6	74.2±1.4	76.8±1.3	77.4±1.2	77.6±1.1	77.8±1.1
75	Classical	61.3±1.7	72.6±1.5	83.8±1.3	85.2±1.2	85.6±1.1	85.8±1.0	85.9±1.0
85	Classical	70.4±1.6	81.4±1.4	91.6±1.2	92.8±1.1	93.1±1.0	93.2±0.9	93.3±0.9
95	Classical	77.8±1.4	88.2±1.2	96.4±1.0	97.1±0.9	97.3±0.8	97.4±0.8	97.4±0.8
65	Ultrasonic	61.2±1.5	72.4±1.4	84.8±1.2	87.3±1.0	87.8±0.9	88.0±0.9	88.1±0.9
75	Ultrasonic	70.6±1.4	82.8±1.2	92.6±1.0	94.1±0.9	94.4±0.8	94.5±0.8	94.6±0.8
85	Ultrasonic	79.3±1.3	89.6±1.1	97.2±0.8	98.1±0.7	98.3±0.6	98.4±0.6	98.4±0.6
95	Ultrasonic	84.6±1.1	93.8±0.9	99.1±0.6	99.4±0.5	99.5±0.5	99.5±0.5	99.5±0.5

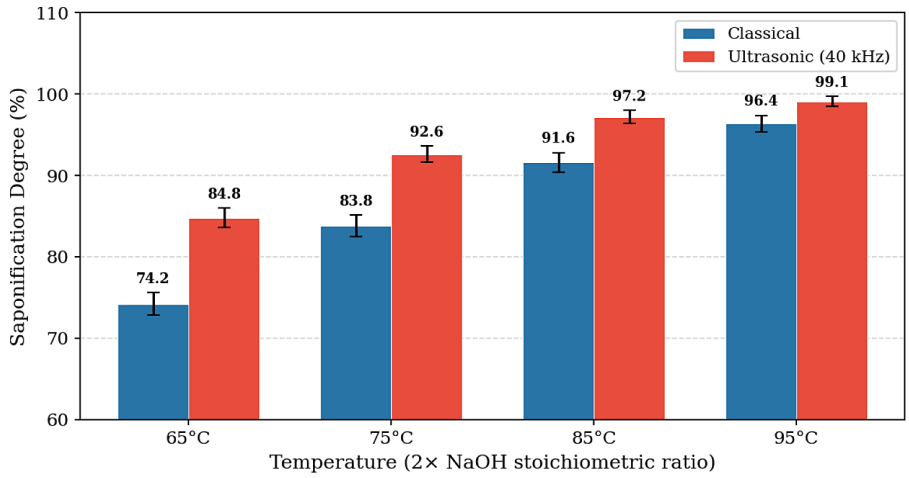

Figure 1. Effect of temperature on saponification degree at 2× NaOH: classical (blue) vs. ultrasonic (red). Error bars = ± SD, n = 3

limited by the high emulsion viscosity, and microjet streaming enhances NaOH access to the substrate at velocities up to 100–200 m/s (Vlachos et al., 2006; Cravotto & Cintas, 2006). At elevated temperatures, thermal diffusion rates are already high and acoustic enhancement is proportionally diminished.

Third, ultrasonic treatment at 85 °C achieved a saponification degree of 97.2%, exceeding that of the classical method at 95 °C (96.4%), representing a 10 °C reduction in operating temperature.

Kinetic analysis (Table 2, Figure 2) demonstrated accelerated conversion under ultrasonic conditions: 90.5% saponification was achieved at 120 min by the ultrasonic method at 85 °C, whereas the classical method at 95 °C required approximately 240 min for equivalent conversion. The ultrasonic method reached a plateau (> 96.9%) at 210 min, compared to 330 min for the classical method – a 36.4% reduction in reaction time (Shamuratov et al., 2025; Shamuratov et al., 2026).

Rheological parameters – viscosity and density

The effects of temperature and NaOH excess on the rheological properties of soapstock during saponification are presented in Tables 3–4 and Figures 3–4.

The initial soapstock exhibited a viscosity of 1.85 ± 0.06 Pa·s, attributable to the high internal friction of the polydisperse emulsion (volume-median drop diameter d_{50} ~7.4 μm). Under optimised ultrasonic conditions (95 °C, 4× NaOH), viscosity decreased to 0.82 ± 0.03 Pa·s, representing a 55.7% reduction. The greatest viscosity reduction occurred between 1× and 2× NaOH: under classical conditions at 85 °C, from 1.51 to 1.24 Pa·s (−17.9%); under ultrasonic treatment at 85 °C, from 1.25 to 1.01 Pa·s (−19.2%). Further increases in NaOH excess had no significant effect on viscosity. Ultrasonic treatment reduced viscosity by 15–22% compared to classical processing at identical temperatures; at 85 °C with 2× NaOH, η = 1.01 Pa·s was achieved (Chemat et al., 2011; Cravotto & Cintas, 2006; Gogate & Pandit, 2004).

Density decreased from 1.462 g/cm³ (65 °C, 1×, classical) to 1.298 g/cm³ (95 °C, 4×, ultrasonic). Micelle formation during saponification traps water molecules, reducing available free water and thereby lowering density (Rosen, 2004). Ultrasonic samples exhibited density values 0.014–0.020 g/cm³ lower than classical counterparts at equivalent temperatures, attributable to gas

Table 2. Saponification kinetics: degree of saponification (%) vs. reaction time under optimal conditions (2× NaOH, n = 3, mean ± SD)

Method	30 min	60 min	90 min	120 min	150 min	180 min	210 min	240 min	300 min	330 min
Classical 95 °C	30.6±1.6	51.6±1.5	66.1±1.4	76.1±1.3	82.9±1.2	87.6±1.1	90.9±1.1	93.1±1.0	95.7±0.9	96.4±0.8
Ultrasonic 85 °C	46.4±1.4	70.8±1.2	83.7±1.1	90.5±1.0	94.0±0.9	95.9±0.8	96.9±0.8	-	-	-

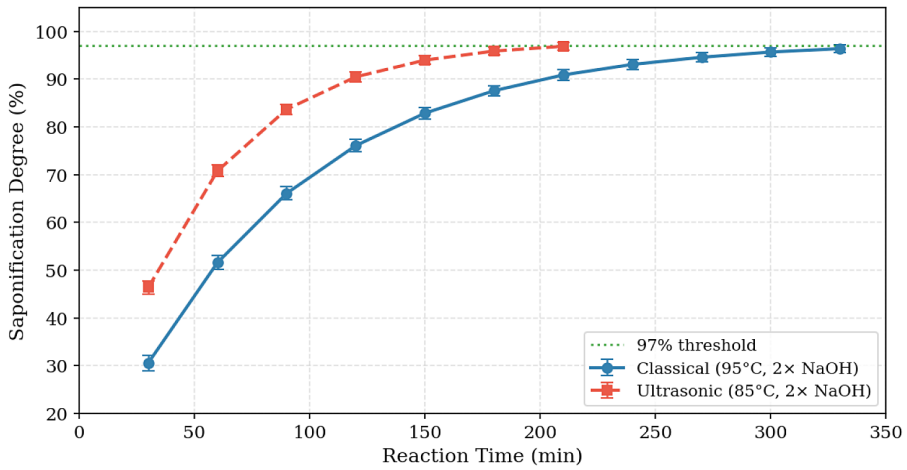

Figure 2. Saponification kinetics: classical method (95 °C, solid circles) vs. ultrasonic 40 kHz (85 °C, dashed squares) at 2× NaOH. Error bars = ± SD, n = 3

Table 3. Dynamic viscosity η (Pa·s, $\gamma = 10\ s^{-1}$, 70 °C, n = 3, mean ± SD) under classical and ultrasonic (40 kHz) conditions

T (°C)	Method	1×	1.5×	2×	2.5×	3×	3.5×	4×
65	Classical	1.85±0.06	1.76±0.05	1.64±0.05	1.61±0.04	1.59±0.04	1.58±0.04	1.58±0.04
75	Classical	1.68±0.06	1.57±0.05	1.44±0.04	1.41±0.04	1.39±0.04	1.38±0.04	1.38±0.04
85	Classical	1.51±0.05	1.39±0.05	1.24±0.04	1.21±0.04	1.19±0.04	1.18±0.04	1.18±0.04
95	Classical	1.32±0.05	1.20±0.04	1.06±0.04	1.04±0.04	1.03±0.04	1.05±0.04	1.05±0.04
65	Ultrasonic	1.57±0.05	1.48±0.05	1.37±0.04	1.34±0.04	1.33±0.04	1.32±0.04	1.32±0.04
75	Ultrasonic	1.42±0.05	1.31±0.04	1.19±0.04	1.16±0.04	1.15±0.04	1.14±0.04	1.14±0.04
85	Ultrasonic	1.25±0.04	1.13±0.04	1.01±0.04	0.98±0.03	0.97±0.03	0.96±0.03	0.96±0.03
95	Ultrasonic	1.08±0.04	0.97±0.04	0.86±0.03	0.84±0.03	0.83±0.03	0.82±0.03	0.82±0.03

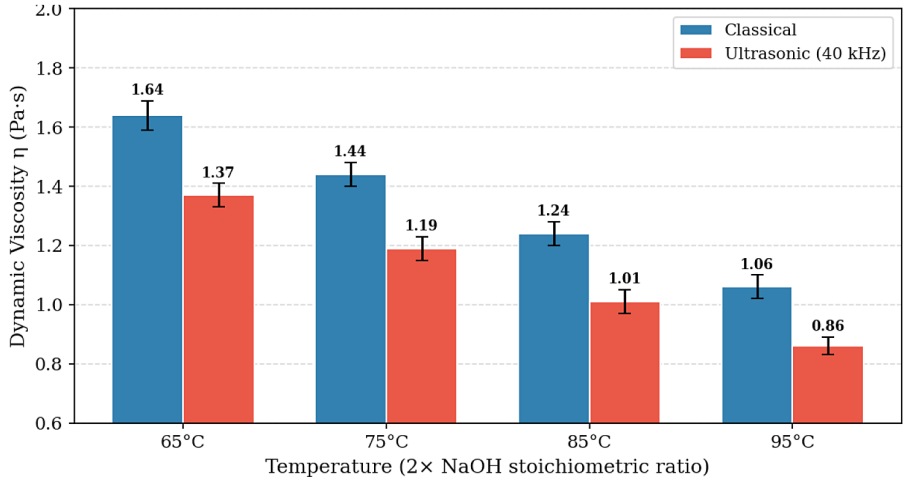

Figure 3. Dynamic viscosity at optimal 2× NaOH: classical (blue) vs. ultrasonic (red). Error bars = ± SD, n = 3

Table 4. Density ρ (g/cm³, pycnometer, 20 °C, n = 3, mean ± SD) under classical and ultrasonic (40 kHz) conditions

T (°C)	Method	1×	1.5×	2×	2.5×	3×	3.5×	4×
65	Classical	1.462±0.008	1.448±0.007	1.428±0.007	1.422±0.006	1.418±0.006	1.416±0.006	1.415±0.006
75	Classical	1.438±0.007	1.422±0.007	1.400±0.006	1.394±0.006	1.390±0.006	1.388±0.005	1.387±0.005
85	Classical	1.412±0.007	1.394±0.006	1.368±0.006	1.362±0.005	1.358±0.005	1.356±0.005	1.355±0.005
95	Classical	1.384±0.006	1.364±0.006	1.336±0.005	1.328±0.005	1.324±0.005	1.320±0.005	1.318±0.005
65	Ultrasonic	1.448±0.007	1.432±0.007	1.412±0.006	1.406±0.006	1.402±0.006	1.400±0.006	1.399±0.006
75	Ultrasonic	1.422±0.007	1.406±0.006	1.382±0.006	1.376±0.006	1.372±0.005	1.370±0.005	1.369±0.005
85	Ultrasonic	1.394±0.006	1.376±0.006	1.348±0.005	1.342±0.005	1.338±0.005	1.336±0.005	1.335±0.005
95	Ultrasonic	1.364±0.006	1.344±0.005	1.316±0.005	1.308±0.005	1.304±0.005	1.300±0.004	1.298±0.004

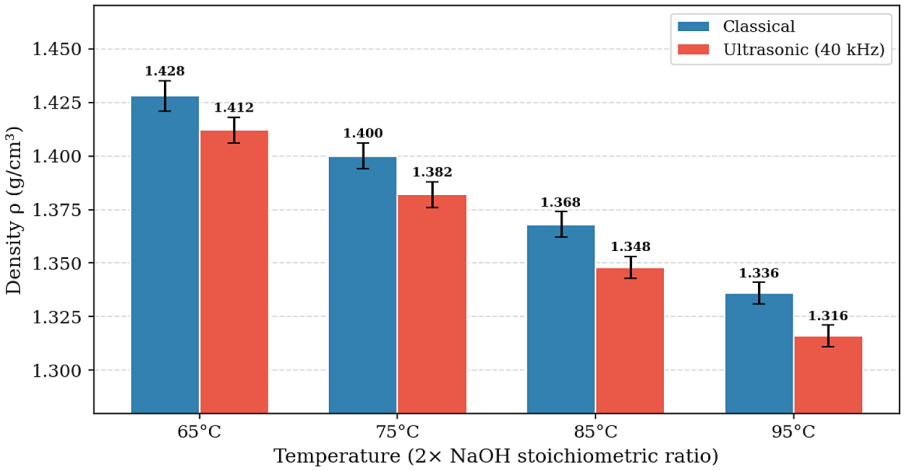

Figure 4. Density at optimal 2× NaOH: classical (blue) vs. ultrasonic (red). Error bars = ± SD, n = 3

microbubble formation and acoustic cavitation-induced homogenisation (Suslick, 1990; Cravotto & Cintas, 2006). A significant inverse Pearson correlation between saponification degree and density was established ($r = -0.91$, $p < 0.001$).

Foaming behaviour

The influence of temperature and NaOH excess on foaming is summarised in Table 5 and Figure 5.

No foaming was observed at 65 °C with either method, consistent with low micellar activity of soapstock components below 70 °C (Rosen, 2004). Ultrasonic treatment reduced foaming across all temperatures studied: at 95 °C, Kf decreased from 0.16 to 0.12 (−25%); at 85 °C, from 0.10 to 0.07 (−30%). On average, ultrasonic treatment reduced foaming by a factor of 1.5–2.5 relative to classical processing. Acoustic cavitation disrupts micellar clusters and redistributes surfactants into the bulk liquid phase rather than allowing accumulation at the liquid-gas interface (Gogate & Pandit, 2004; Cravotto & Cintas, 2006). The 56.3% reduction in Kf under optimal conditions substantially simplifies foam management in subsequent salting-out and acid decomposition stages, improving volumetric process efficiency.

FTIR Spectroscopic Analysis

Raw cotton soapstock

FTIR absorptions of raw soapstock are presented in Table 6 and Figure 6. The broad O-H stretch at 3397.96 cm^{-1} (transmittance 14.16%) confirms the presence of hydrogen-bonded moisture, consistent with the 40-60% water content characteristic of soapstock (Haas, 2005). Strong C-H stretching bands at 2940.91 and 2863.77 cm^{-1} are indicative of methylene groups from long-chain (C16-C18) fatty acids, typical of cottonseed oil (Guillén & Cabo, 1997; Vlachos et al., 2006; Christy et al., 2019). The band at 1735.62 cm^{-1} corresponds to the C=O ester stretch of triglycerides, representing the primary substrate for saponification (Guillén & Cabo, 1997; Guillén &

Table 5. Foaming properties of saponified soapstock under classical and ultrasonic (40 kHz) conditions (n = 3)

T (°C)	Method	H_0 (cm^3)	H_5 (cm^3)	H_0/H_5	Kf (t=0)	Kf (5 min)	Stability
65	Classical	-	-	-	-	-	No foam
75	Classical	105	102	1.03	0.05	0.02	Moderate
85	Classical	110	106	1.04	0.10	0.06	Moderate
95	Classical	116	111	1.05	0.16	0.11	High
65	Ultrasonic	-	-	-	-	-	No foam
75	Ultrasonic	102	100	1.02	0.02	0.00	Low
85	Ultrasonic	107	104	1.03	0.07	0.04	Low
95	Ultrasonic	112	109	1.03	0.12	0.09	Low

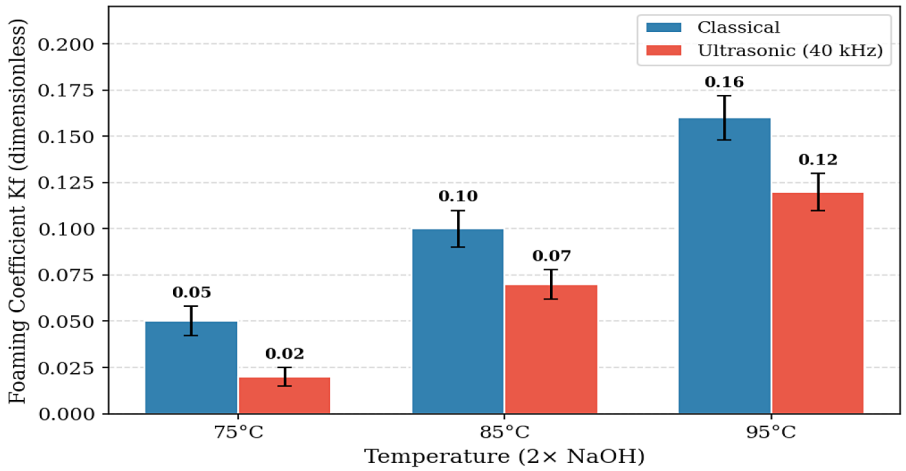

Figure 5. Foaming coefficient Kf (t = 0) at 2× NaOH: classical (blue) vs. ultrasonic (red). Error bars = ± SD, n = 3. No foam observed at 65 °C

Table 6. FTIR band assignments for raw cotton soapstock (ATR mode, 400–4000 cm^{-1})

Wavenumber (cm^{-1})	%T	Assignment	Compound class
3397.96	14.16	O-H stretch (broad, H-bonded)	FFA / moisture
2940.91	75.20	C-H asym. stretch (-CH_2-, -CH_3)	Long-chain alkyl
2863.77	81.89	C-H sym. stretch (-CH_2-)	Long-chain alkyl
1735.62	97.51	C=O stretch (ester carbonyl)	Triglycerides / esters
1644.98	69.33	C=C stretch + O-H bend	Unsaturated FA + H_2O
1548.56	86.06	N-H deformation / COO^-	Protein impurities
1419.35	92.40	C-H scissoring (-CH_2-)	Methylene chains
1147.44	99.54	C-O-C asym. stretch	Ester linkage
1064.51	98.74	C-O stretch	Ester / alcohol
752.10	80.55	=C-H out-of-plane bend	Unsaturated alkyl

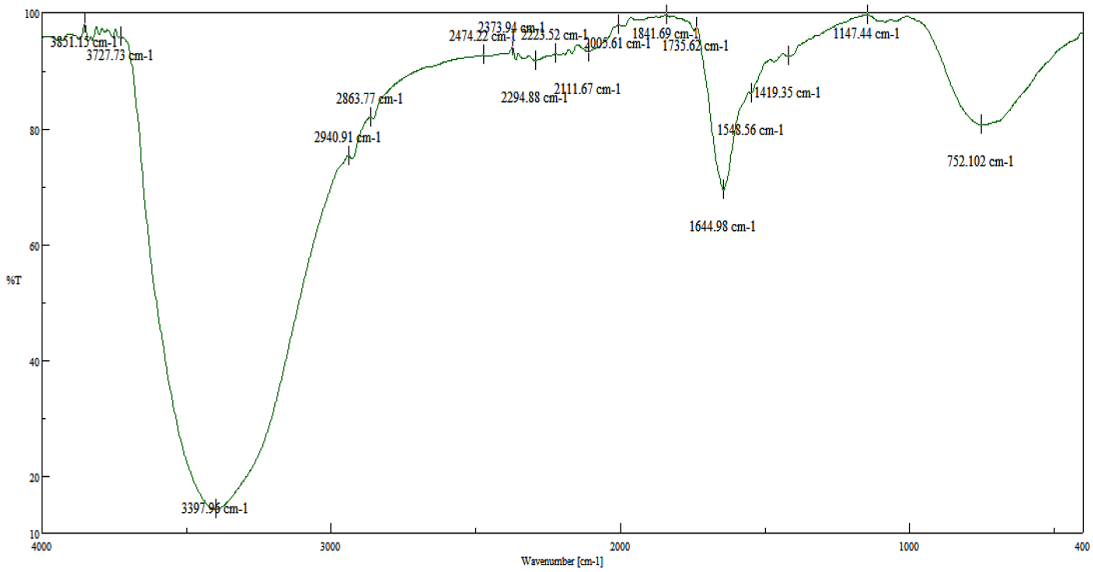

Figure 6. FTIR spectrum of raw cotton soapstock (ATR mode)

Cabo, 1998). The C=C stretch at 1644.98 cm^{-1} is characteristic of linoleic acid (C18:2), a principal component of cottonseed oil (Christy et al., 2019). The C-O-C asymmetric stretch at 1147.44 cm^{-1} confirms intact ester linkages in the unsaponified material (Lerma-García et al., 2009; Rohman & Che Man, 2011).

Saponified cotton soapstock

The most diagnostic spectral change following saponification was the near-complete disappearance of the ester C=O band at ~1735 cm^{-1} and the emergence of a strong asymmetric COO^- stretch at 1603.52 cm^{-1} (transmittance 84.12%), marking conversion of esterified fatty acids to sodium carboxylate soaps (Table 7, Figure 7) (Lerma-García et al., 2009; Deacon & Phillips, 1980; Vlachos et al., 2006).

The symmetric COO^- stretch at 1420.32 cm^{-1} (transmittance 72.71%) was also evident. The band separation ($\Delta\nu$ = 183 cm^{-1}) between the asymmetric and symmetric stretches corresponds to bidentate coordination expected in sodium salts of fatty acids (Deacon & Phillips, 1980; Vlachos et al., 2006). The C-H stretching bands at 2921.63 cm^{-1} (40.72%) and 2852.20 cm^{-1} (59.28%) increased substantially in intensity relative to raw soapstock (75-82%), consistent with structural reorganisation from buried ester-bound alkyl chains to exposed soap molecule chains (Guillén & Cabo, 1997; Lerma-García et al., 2009; Christy et al., 2019). Disappearance of the C-O-C ester stretch at ~1147 cm^{-1} confirmed complete hydrolysis of ester bonds (Lerma-García et al., 2009; Deacon & Phillips, 1980). The FTIR results are fully consistent with the titrimetric data, corroborating a saponification degree of 97.2% (Guillén & Cabo, 1997; Deacon & Phillips, 1980).

Table 7. FTIR band assignments for saponified soapstock (US, 85 °C, 2× NaOH, ATR mode)

Wavenumber (cm^{-1})	%T	Assignment	Structural significance
3628.41	78.22	O-H stretch (free carboxylic)	Soap / FFA
3399.89	36.55	Broad O-H stretch (H-bonded)	Hydrated soap micelles
2921.63	40.72	C-H asym. stretch	Exposed alkyl chains (soap)
2852.20	59.28	C-H sym. stretch	Methylene (soap)
1603.52	84.12	Asym. COO^- stretch	Sodium carboxylate (soap)
1557.24	65.13	Asym. COO^- / N-H def.	Carboxylate anion
1420.32	72.71	Sym. COO^- stretch	Soap - confirms saponification
1149.37	98.87	C-O remnant	Residual ester traces
700.998	87.74	=C-H bending	Unsaturated alkyl

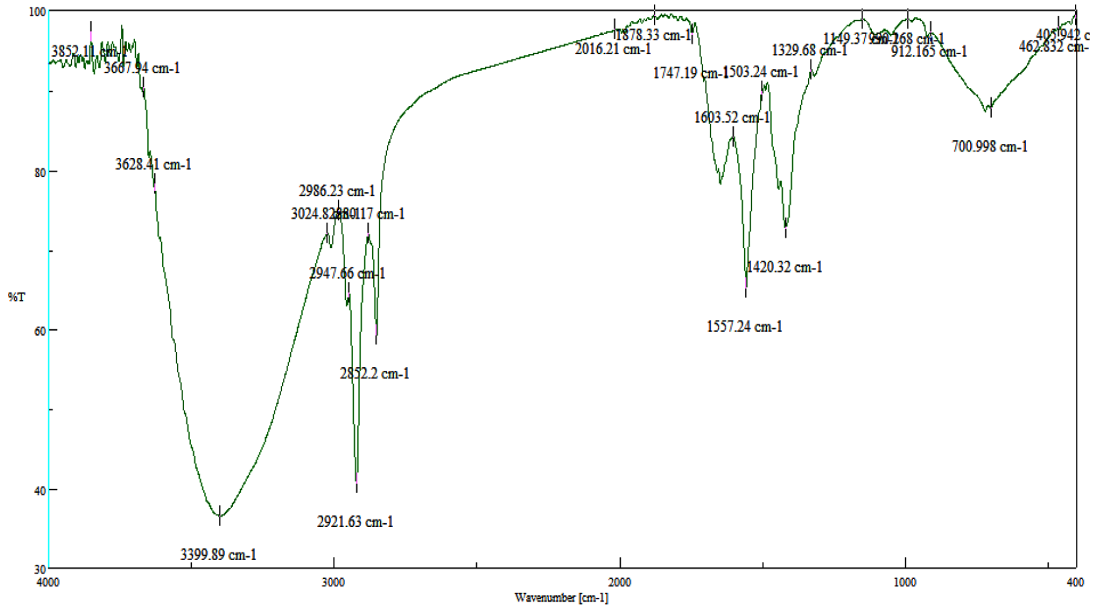

Figure 7. FTIR spectrum of saponified soapstock (US, 85 °C, 2× NaOH, ATR mode). Key COO^- formation bands annotated

Integrated optimisation and process implications

Table 8 demonstrates that ultrasonic treatment at 85 °C with 2× NaOH simultaneously achieved all four process targets: saponification degree 97.2% (threshold 97%); dynamic viscosity 1.01 Pa·s (threshold 1.05 Pa·s); density 1.348 g/cm³ (threshold 1.35 g/cm³); and foaming coefficient 0.07, substantially below the classical value of 0.10.

Strong inverse correlations were observed between saponification degree and viscosity ($r = -0.94$, $p < 0.001$), and between saponification degree and density ($r = -0.91$, $p < 0.001$), indicating internal consistency of the measurements and confirming that observed rheological changes reflect genuine chemical transformation rather than physical mixing effects (Mezger, 2014; Rao, 2010). FTIR results corroborate these findings.

For a facility processing 100 tonnes of soapstock per day, reducing the saponification temperature from 95 °C to 85 °C yields 8–12% steam savings per batch (Chemat et al., 2011). The 2× NaOH protocol also reduces reagent consumption and downstream waste. These outcomes align with circular economy objectives for the oleochemical sector, enabling valorisation of refinery waste streams with reduced environmental impact (Bhosle & Subramanian, 2005; Haas, 2005).

Table 8. Comparative performance: classical (95 °C, 2× NaOH) vs. ultrasonic (85 °C, 2× NaOH) saponification

Parameter	Classical 95 °C, 2×	Ultrasonic 85 °C, 2×	Change
Saponification degree (%)	96.4±1.0	97.2±0.8	+0.8 p.p.
Viscosity η (Pa·s, 70 °C)	1.06±0.04	1.01±0.04	−4.7%
Density ρ (g/cm³, 20 °C)	1.336±0.005	1.348±0.005	−1.5% at 85 °C
Foaming coefficient Kf	0.16	0.07	−56.3%
C=O ester band (cm^{-1})	1735 (present)	1735 (absent)	Fully saponified
COO^- stretch (cm^{-1})	Weak 1603	Strong 1603 (%T 84.1)	Complete conversion
Operating temperature	95 °C	85 °C	−10 °C (energy saving)

CONCLUSIONS

Ultrasonic saponification at 40 kHz represents an effective and sustainable strategy for CFA recovery from cotton soapstock waste. The principal findings of this study are as follows:

1. Ultrasonication at 85 °C with 2×NaOH achieved a saponification degree of 97.2%, matching or exceeding the classical method at 95 °C (96.4%), with a 10 °C reduction in operating temperature corresponding to 8–12% energy savings.
2. Across all four rheological parameters, 2× NaOH was identified as the optimal stoichiometric ratio, with higher excesses yielding no statistically significant improvement.
3. Ultrasonic treatment reduced dynamic viscosity by 15–22% relative to classical processing, attributable to acoustic cavitation-induced emulsification, which decreased droplet size from 7.4 to 2.3 µm and enhanced mass transfer efficiency in downstream processing.
4. Ultrasonic conditions reduced the foaming coefficient by a factor of 1.5–2.5, substantially simplifying foam management in downstream acidulation and phase separation.
5. FTIR spectroscopic analysis provided definitive chemical evidence of complete saponification, with disappearance of the ester carbonyl band at 1735.62 cm^{-1} and emergence of the carboxylate stretch at 1603.52 cm^{-1}, in full agreement with titrimetric results.
6. The integration of rheological and FTIR data provides a robust multi-parameter analytical framework for monitoring process quality in industrial-scale CFA recovery from diverse oilseed processing waste streams.

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