



RESEARCH ARTICLE

Synthesis, Spectral and Thermo-Gravimetric Analysis of Novel Macromolecular Organo-Copper Surfactants

Anju Joram¹, Rashmi Sharma¹ and Arun K. Sharma^{2,*}

¹Department of Chemistry, S.P.C. Govt. College Ajmer-305001, Rajasthan, India

²Department of Chemistry, Govt. P.G. College, Jhalawar-326001, Rajasthan, India

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Abstract:**Background:**

The present paper highlights:

Synthesis of copper surfactants derived from edible oils *i.e.* Groundnut & Sesame and non-edible oils *i.e.* Neem & Karanj.

Methods:

Spectral studies (IR, NMR) have been carried out to understand the structural insight of the surfactants synthesized.

Results and Conclusion:

Thermogravimetric analysis of copper surfactants derived from Groundnut, Sesame, Neem & Karanj has been done to confirm the thermal decomposition/stability. Kinetic parameter *i.e.* activation energy and thermodynamic parameters *i.e.* Gibbs free energy, entropy and enthalpy were calculated by five different well-known equations namely Freeman Carroll, Coats - Redfern, Horowitz - Metzger, Broido, and Piloyan - Novikova.

Keywords: Copper surfactants, IR, NMR, ESR, TGA, Gibbs free energy.

1. INTRODUCTION

Artificial and Natural surfactants or surface active agents are materials that tend not only to accumulate at surfaces but also their presence change the properties of earth and other surfaces. Surfactants are one such group of compounds which are used in various fields of science [1]. Surface active agents are vital components in biological systems, form key ingredients in consumer products and play an important role in many industrial processes [2]. Sesame oil is equally important to nurture the external system, mainly the skin and joints. Its effectively treats skin rashes and sores, improves blood circulation and keeps the body warm. Sesame oil (*Sesamum indicum*) has some laxative properties and is also used for cooking because of its high protein and mineral content [3]. Recently, work on poly-metallic complexes and transition metal complexes of heterocyclic ligands has been done; and their structure and biological characteristics have also been discussed [4]. Various workers have studied different spectral technique like IR, ESR, NMR and magnetic moment studies of various complexes containing Copper soaps to determine their geometrical and structural aspects [5, 6]. Neem (*Azadirachta indica*) and karanj (*Pongamia pinnata*) are very good for Anthelmintic (parasites and worms), and anti-inflammatory properties [7]. The most characteristic property of amphiphilic molecules is the capacity to aggregate in solutions. A number of papers have been reported to elaborate and analyse the micellar features of copper (II) soaps by density studies [8], apparent molar volume [9], viscosity [10], surface tension [11], parachor [12] ultrasonic [13, 14] and electrical conductance [15]. Present work has been initiated with a view to obtain a profile

* Address correspondence to this author at the Department of Chemistry, Govt. P.G. College, Jhalawar-326001, Rajasthan, India; Tel: +919352669899; E-mail: sharmaarun423@gmail.com

regarding structural insight of thermal decomposition of Copper-Groundnut Surfactant (CG), Copper-Sesame Surfactant (CS), Copper-Neem Surfactant (CN), and Copper-Karanj Surfactant (CK). Thermal degradation and use of various equations will provide valuable information about the kinetics of the degradation reaction [16]. Thermal stability, kinetic parameters, solute-solvent and solute-solute interactions, photo-catalytic degradation [17] and other fundamental studies will play a significant role in the selection of synthesized copper surfactants.

2. EXPERIMENTAL

2.1. Synthesis

Groundnut, Sesame, Neem and Karanj oils were extracted from their Kernels respectively using petroleum ether and then purified. The fatty acid composition of these edible and non-edible oils (Table 1) was confirmed through GLC of their methyl esters. Copper (II) surfactants were prepared from edible oils *i.e.* Groundnut (*Acharishypogaea*) and Sesame oil (*Sesamum indicum*) and non-edible oils *i.e.* Neem (*Azadirachta indica*) and Karanj (*Pongamia pinnata*) oils by Direct Metathesis process [18]. The physical and analytical properties have been mentioned in Table 2.

Table 1. Fatty acid composition of oils used for copper surfactants synthesis.

Name of Oils	% Fatty Acids				
	16:0	18:0	18:1	18:2	Other Acids C ₂₀ -C ₂₄
Groundnut Oil	10	4	61	18	7
Sesame Oil	8	4	45	41	-
Neem Oil	14.9	14.4	61.9	7.5	1.3
Karanj Oil	5.2	5.0	57.3	13.8	18.7

Table 2. Analytical and physical data of copper surfactant derived from edible and non-edible oils.

Name of Soap	Colour	M.P. (°C)	Metal Content		S.V.	S.E.	Av. M. W.
			Observed	Calculated			
CG	Dark Green	98	10.15	9.72	188.70	297.29	658.08
CS	Dark Green	104	10.05	9.87	175.80	319.11	701.72
CN	Dark Green	50	10.36	10.11	198	283.33	630.16
CK	Dark Green	51	9.49	9.34	181.5	309.09	681.68

2.2. Instrumentation

In order to study surfactants and reconfirmation of the synthesized molecule, the Infra-red absorption spectra of four compounds derived from edible and non-edible oils were obtained on a FTIR-spectrophotometer, (Shimadzu 821PC (4000 - 400 cm⁻¹) and Perkin Elmer infrared spectrophotometer from Sophisticated Analytical Instrument Facility, CDRI, Lucknow. Proton NMR spectra were recorded at SAIF, CDRI, Lucknow on NMR spectrometer, Bruker DRX-300 at 300 K using C₆D₆ (deuterated benzene) as solvent for surfactants. The samples were sent to SAIF, IIT-Powai, Mumbai for Thermogravimetric analysis. The TGA curves of the above mentioned compounds were obtained by Perkin Elmer Thermal Analysis apparatus. TGA was done on Nitrogen (N₂) atmosphere between 0°C-600°C at the rate of 10°C per minute. The results were obtained as plots of 'weight loss v/s temperature' and '%weight loss v/s temperature'.

3. CHARACTERIZATION

3.1. IR Spectral Studies

The detailed infrared absorption spectral studies revealed that there is a marked difference between the spectra of

oils and that of corresponding copper surfactant of four copper soaps synthesized for the study [19 - 20]. The details of IR spectral peaks are summarized in Table 3.

Table 3. IR spectral data of copper surfactants.

Absorption Bands	CG cm ⁻¹	CB cm ⁻¹	CN cm ⁻¹	CK cm ⁻¹
CH ₃ and CH ₂ , C-H Anti symm. stretching (v _{as})	2975-2960	2975-2953	2925.6	2927.6
CH ₃ and CH ₂ , C-H Symm. Stretching (v _s)	2850-2840	2855-2840	2855.1	2855.7
>C=O stretching	-	-	1727.6	1736.0
COO ⁻ , C-O Antisymm. Stretching	1590	1590	1592.8	1609.3
CH ₃ , C-H sym. Bending (δ _s)	-	-	1377	1377.3
CH ₂ , C-H Bending (δ) (twisting and wagging)	1295	1310	1340	1345
=C-H, out-of-plane bending of C-H (strong)	-	-	761.3	759.1
CH ₂ , C-H rocking	730	730	670.2	669.2
Cu-O stretching	665	670	480	480

3.2. NMR Spectral Studies

The NMR spectra used to confirm the nature and structure of copper soaps synthesized for the study [21, 22]. The details of NMR signals are summarized in Table 4.

Table 4. NMR spectral data for copper surfactants.

Soaps Assignments	CG (δ)	CS (δ)	CN (δ)	CK (δ)
-CH ₃ -CH ₂ -R	0.91	0.97	0.904	0.918
-CH ₂ -CH ₂ -R	1.28	1.293	1.277	1.229
-CH ₂ -C=C-	2.10	2.062	2.042	2.093
-C=C-H (vinyllic proton)	5.48	5.47	5.432	5.254
-CH ₂ -C(=O)OCu	2.229	2.233	-	-

4. RESULTS AND DISCUSSION

4.1. Thermal Decomposition

The thermogravimetric analysis of copper (II) soaps derived from mustard oil and soybean oil have been done earlier by Sharma. *et al.* [23]. The thermal decomposition profiles for CN, CK, CG and CS (Figs. 1-4), where all the TGA curves have three folds single decomposition step in the range of 423 to 688 K (150 to 500°C). The copper surfactants then finally decompose into parent ketones, Cu₂O and CO₂ [24].

Thermal decomposition of these surfactants derived from groundnut, sesame, neem and karanj oil occurred in three stages, related to the decomposition of polyunsaturated, monounsaturated and saturated fatty acids, respectively [25]. In relation to the thermal decomposition three folds but single step, it was observed that the first fold (150°C to 220°C) 423K to 493K corresponds to the decomposition of the polyunsaturated fatty acids. The second fold in the thermal decomposition of soaps derived from edible and non-edible oils have been observed in the range of 483K to 568K (210°C to 295°C) corresponds to the decomposition of mono unsaturated fatty acids. The third fold in the thermal decomposition, which occurs in the temperature range of 553K to 75K (280°C to 478°C), corresponds to the thermal decomposition of the saturated fatty acids.

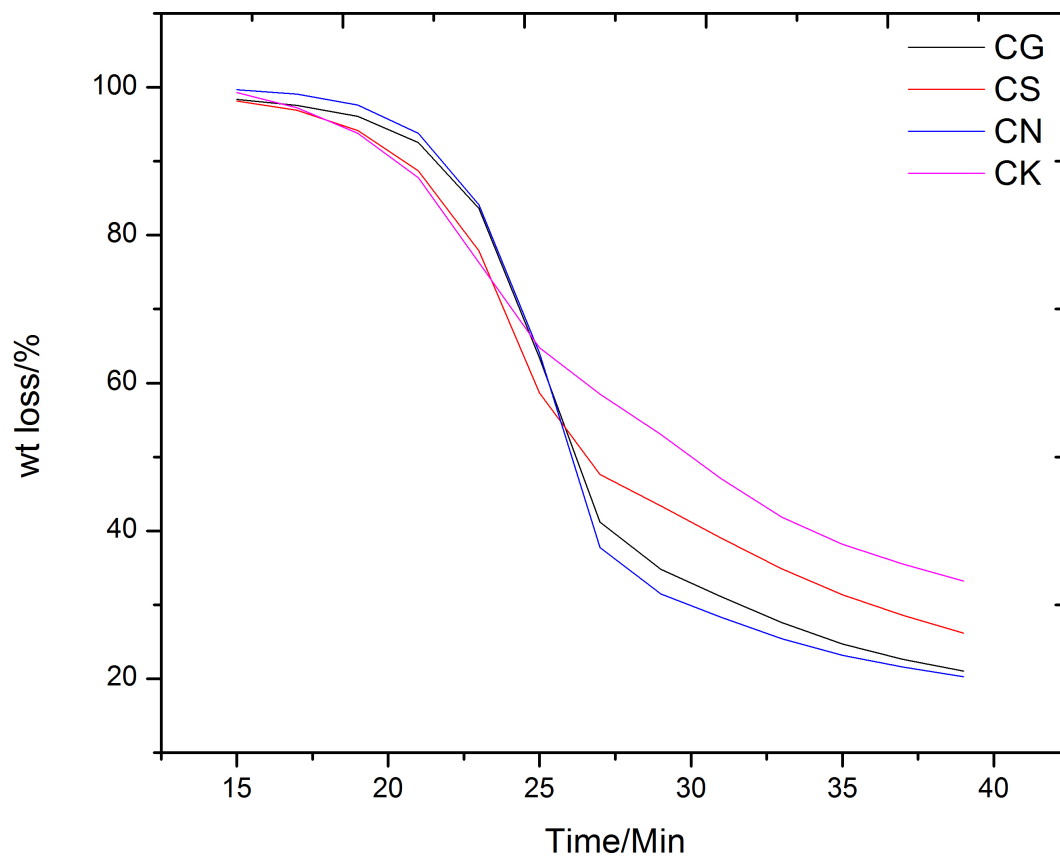


Fig. (1). Thermogram of % wt loss v/s time for copper surfactants derived from various oils.

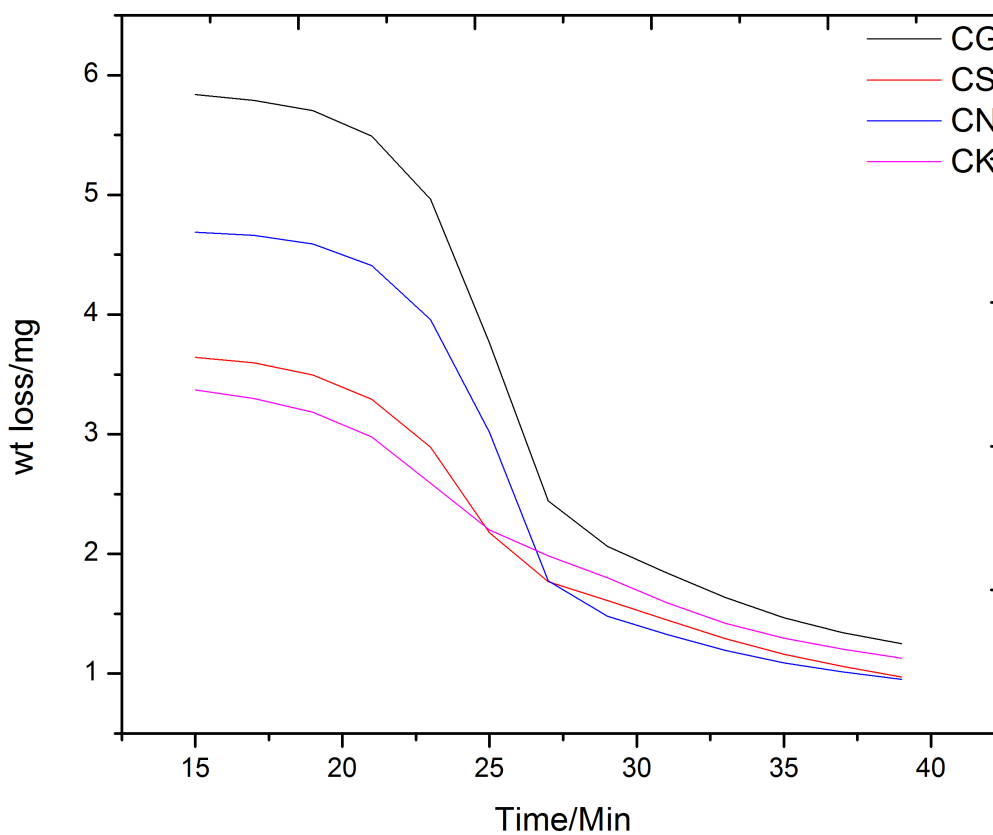


Fig. (2). Thermogram of wt loss v/s time for copper surfactants derived from various oils.

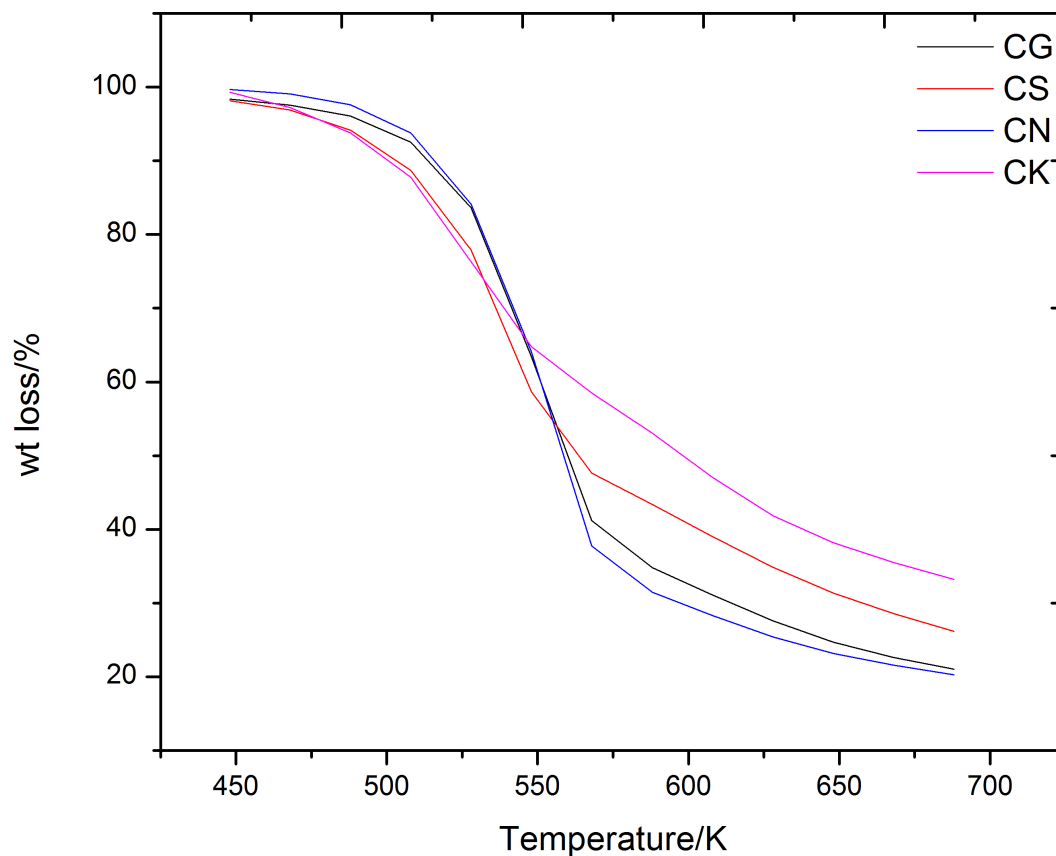


Fig. (3). Thermogram of % wt loss v/s temperature for copper surfactants derived from various oils.

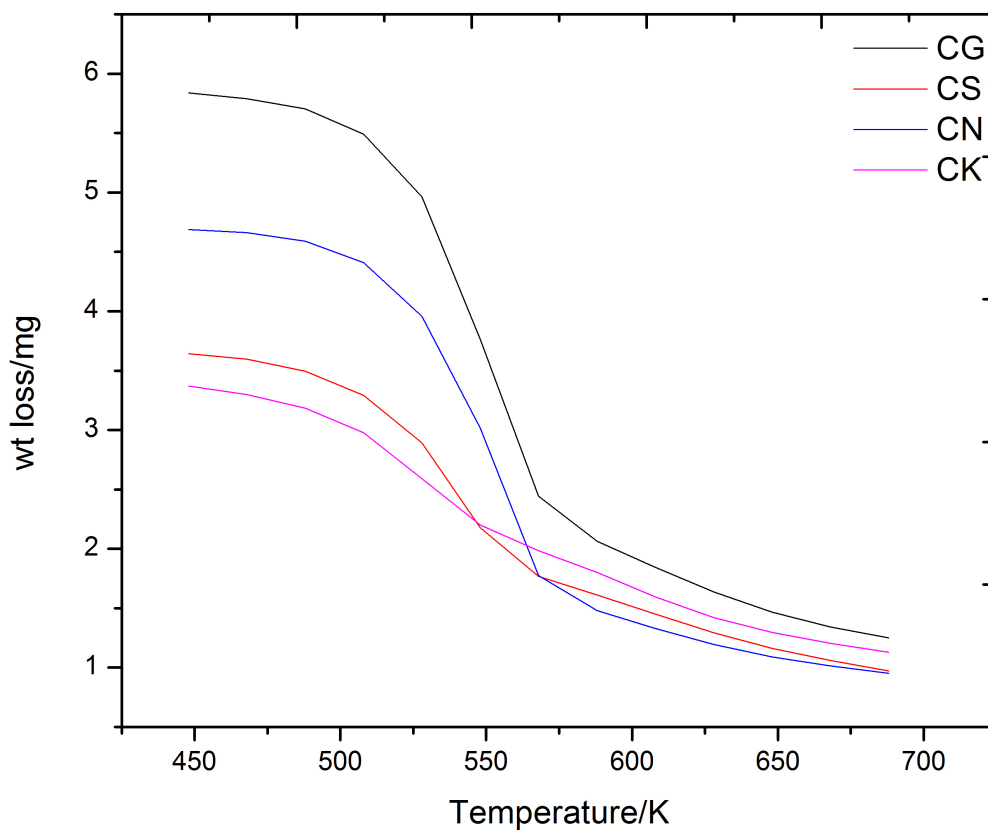


Fig. (4). Thermogram of wt loss v/s temperature for copper surfactants derived from various oils.

In our referred systems, *i.e.* CG, CS, CN and CK have almost same thermal stability as the first fold thermal decomposition begins at 423K in all four the cases. An interesting observation noted on perusal of results is that the first fold for CG lies in the range of 427K to 483K (150°C to 210°C), CS lies in the range of 423K to 473K (150°C to 200°C), CN lies in the range of 428K to 488K (155°C to 215°C) and CK lies in the range of 423K to 485K (150°C to 212°C). Hence, the possibility cannot be denied that CN, which is derived from neem oil, takes longer time and required higher temperature to completely decompose its Polyunsaturated Fatty Acid (PUFA) content as compared to CG, CS and CK, which have been derived from groundnut, sesame and karanj oil. respectively Similarly, for second fold decomposition of CG the range lies in 483K to 563 K (210°C to 290°C), for CS the range lies in 473K to 543K (200°C to 210°C), for CN the range lies in 488K to 568K (215°C to 295°C) and for CK the range lies in 485K to 565K (212°C to 292°C). Thus, again it may be suggested that CN needs longer time and higher temperature to completely decompose its Monounsaturated Fatty Acid (MUFA) content as compared to CG, CS and CK, which have been derived from groundnut, sesame and karanj oil. respectively. For the third fold, for CG the decomposition range lies between 563K to 703K (290°C to 430°C), for CS the range lies between 543K to 673K (270°C to 400°C), for CN the range lies between 568K to 751K (295°C to 478°C) and for CK the range lies between 565K to 748K (292°C to 475°C). Therefore, it can be suggested that CN (derived from Neem oil) needs longer time and higher temperature to completely decompose its saturated fatty acid content as compared to CG, CS and CK, which are derived from groundnut, sesame and karanj oil. respectively. Thus, the possibility cannot be denied that CN possesses higher thermal stability in comparison to CG, CS and CK.

4.2. Kinetic Parameters

All these results have been applied in various equations like Freeman Carroll [26] Coats-Redfern equation [27], Horowitz-Metzger equation [28], Broido [29] and Piloyan–Novikova Equation [30] to evaluate the energy of activation (E) for thermal degradation of the single step of these surfactants.

Freeman Carroll equation given as follows:

$$\ln\left(\frac{dw}{dt}\right)W_r = \ln Z - \frac{E}{RT} \quad (1)$$

The value of energy of activation using F-C equation for each step was evaluated from the plots of $\ln[-\ln(1-\alpha)]$ v/s $1/T$ (Fig. 5).

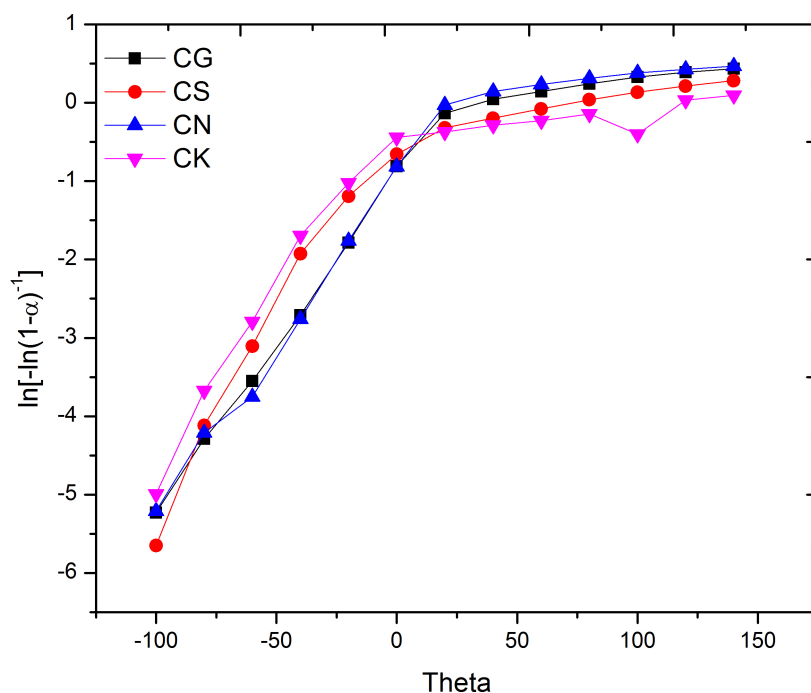


Fig. (5). Plot of Freeman Corrolls equation $\ln(dw/dt)/dw_r$ v/s $1/T$ for copper surfactants derived from various oils.

From this equation, the values of energy of activation are observed to be in the following order for CG, CS, CN and CK.

CG > CS (for edible oils)

CN > CK (for non-edible oils)

Coats and Redfern derived the following equation:

$$\frac{\log[-\log(1-\alpha)]}{T^2\alpha E} = \frac{\log AR}{E(1-2RT)} - \frac{E}{2.303RT} \tag{2}$$

The values of energy of activation using Coats-Redfern equation for the step were evaluated from the plots of $\log\{-\log(1-\alpha)\}T^2$ v/s $1/T$ (Fig. 6).

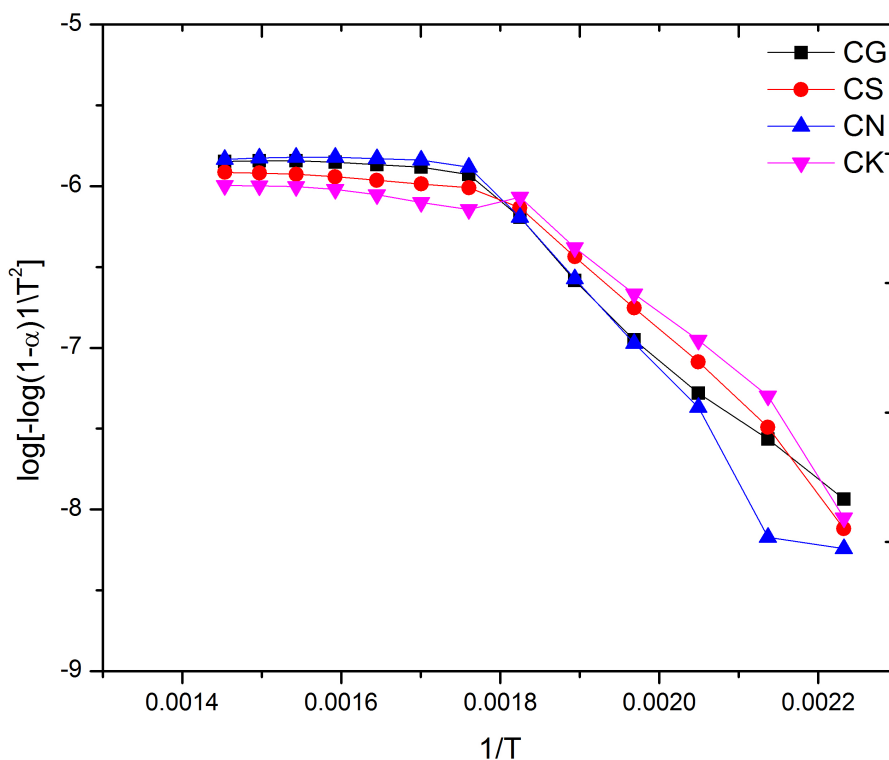


Fig. (6). Plot of Coats and Redfern equation $\log[-\log(1-\alpha)/T^2]$ v/s $1/T$ for copper surfactants derived from various oils.

The values of activation energies evaluated from the slope of these plots are recorded in Table 5. The values of energy of activation are observed to be in the following order for CG, CS, CN and Ck.

CG > CS (for edible oils)

CN > CK (for non-edible oils)

Table 5. Kinetic parameter, energy of activation for the decomposition reaction of copper surfactants derived from various oils.

Compound	Method	E_a	Rate Constant, k (s^{-1})	Decomposition Temperature (K)	Order of Reaction	R^2
		($kJ\ mol^{-1}$)			(n)	
CG	F.C.E.	56.30	1.17×10^{-6}	548	1	0.99
	C.R.E.	54.39	4.22	548	1	0.98
	H.M.E.	56.66	169.11	548	1	0.97
	B.E.	63.77	10.83	548	1	0.99
	P.N.E.	44.67	0.141	548	1	0.98

(Table 5) contd....

Compound	Method	E_a	Rate Constant, k (s^{-1})	Decomposition Temperature (K)	Order of Reaction	R^2
		($kJ\ mol^{-1}$)			(n)	
CS	F.C.E.	48.26	6.6×10^{-6}	528	1	0.99
	C.R.E.	48.48	6.9×10^{-4}	528	1	0.98
	H.M.E.	36.32	9.17	528	1	0.97
	B.E.	37.87	3.88	528	1	0.99
	P.N.E.	35.11	1.6×10^{-2}	528	1	0.98
CN	F.C.E.	62.25	3.1×10^{-7}	548	1	0.99
	C.R.E.	60.87	9.2×10^{-3}	548	1	0.98
	H.M.E.	67.46	62.88	548	1	0.97
	B.E.	62.22	6.27	548	1	0.99
	P.N.E.	70.25	0.294	548	1	0.98
CK	F.C.E.	42.95	2.0×10^{-5}	548	1	0.99
	C.R.E.	44.68	2.8×10^{-4}	548	1	0.98
	H.M.E.	42.91	8.32	548	1	0.97
	B.E.	34.07	3.06	548	1	0.99
	P.N.E.	28.96	4.4×10^{-3}	548	1	0.98

To confirm the energy of activation, Horowitz-Metzger equation was used to evaluate the value of 'E'. The Horowitz-Metzger equation is as follows:

$$\ln[\ln(1-\alpha)-1] = \frac{E\theta}{RT_s^2} \quad (3)$$

Where ' α ' is the fraction of soap decomposed at time ' t ', ' T_s ' is the temperature at which the rate of decomposition is maximum and ' θ ' is equal to $(T-T_s)$. The energy of activation was obtained from the slope of the plot between ' $\ln[\ln(1-\alpha)^{-1}]$ ' v/s ' θ ' (Fig. 7).

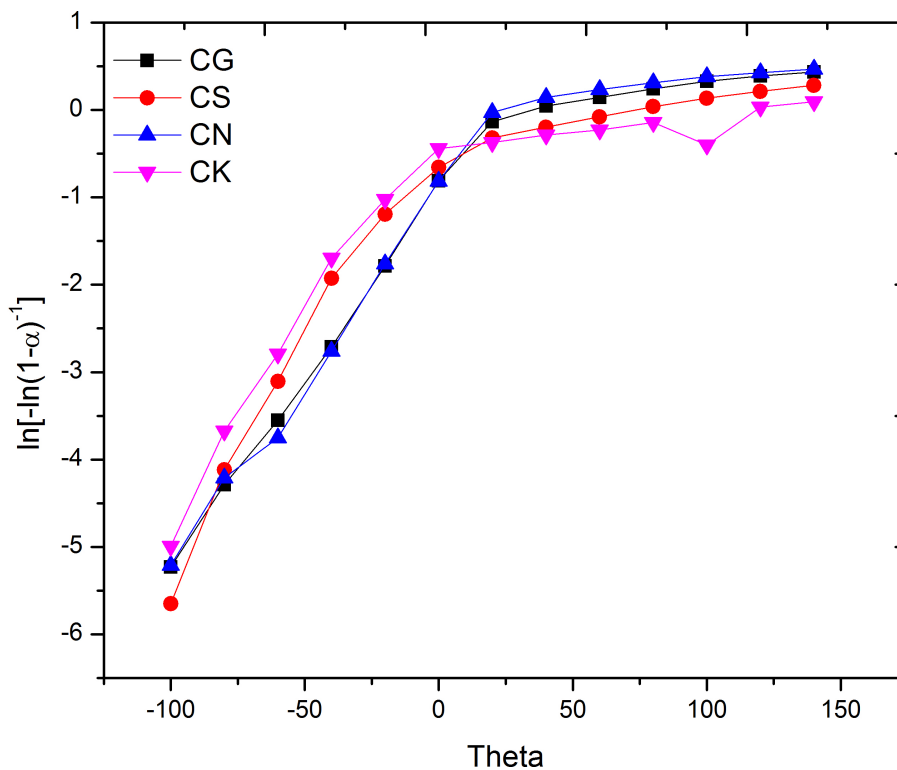


Fig. (7). Plot of Horowitz – Metzger equation $\ln[-\ln(1-\alpha)^{-1}]$ v/s theta for copper surfactants derived from various oils.

For Horowitz-Metzger equation the values of Ea for soaps are in the following order:

CG > CS (for edible oils)

CN > CK (for non-edible oils)

The values of energy of activation for the soaps thermal decomposition of CG, CS, CN and CK were calculated by using Broido's equation which as follows:

$$\ln[\ln(1/y)] = -E + \frac{C}{RT} \tag{4}$$

Where 'y' is fraction of weight at temperature 'T', 'E' is the activation energy and 'R' is the gas constant in J mol⁻¹K⁻¹. The energy of activation for each soap is calculated from the slope of plot between 'ln[ln(1/y)] and (1/T)' as depicted in Fig. (8). The values of activation energies for different soaps of thermal decomposition of CG, CS, CN and CK are recorded in Table 5 and are found to be in the following order:

CG > CS (for edible oils)

CN > CK (for non-edible oils)

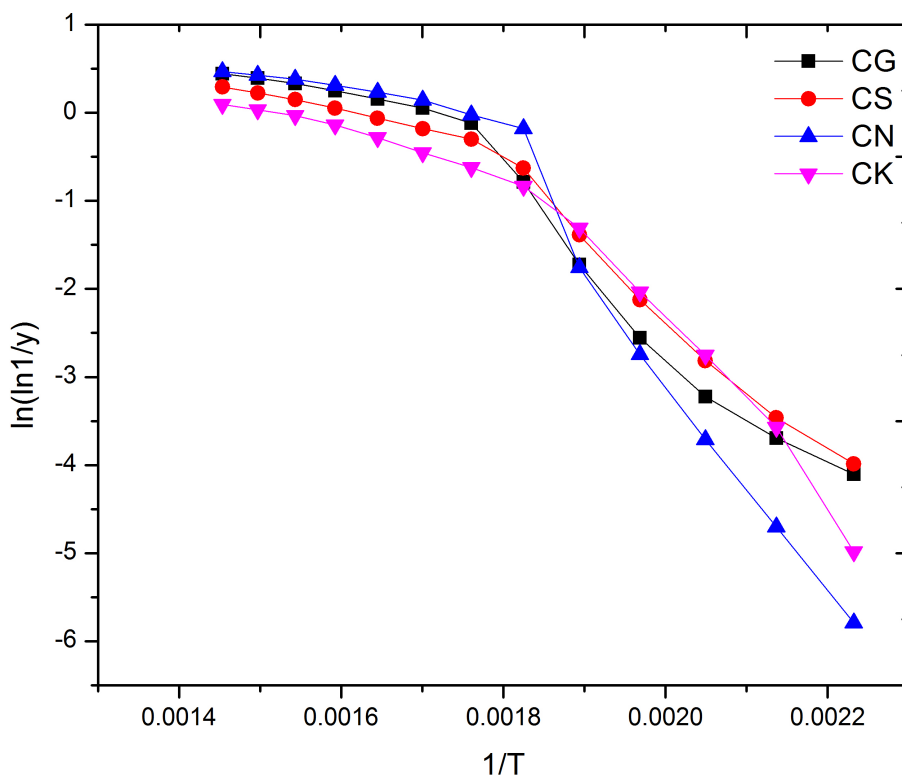


Fig. (8). Plot of Broidoequation ln[ln(1/y) v/s 1/T for copper surfactants derived from various oils.

Piloyan–Novikova Equation is given as follows:

$$\ln[g(\alpha)T^2] = \ln \frac{ZR}{\beta E} - \frac{E}{RT} \tag{5}$$

The values of energy of activation using P-N equation for each step were evaluated from the plots of 'ln(α/T²) v/s 1/T (Fig. 9).

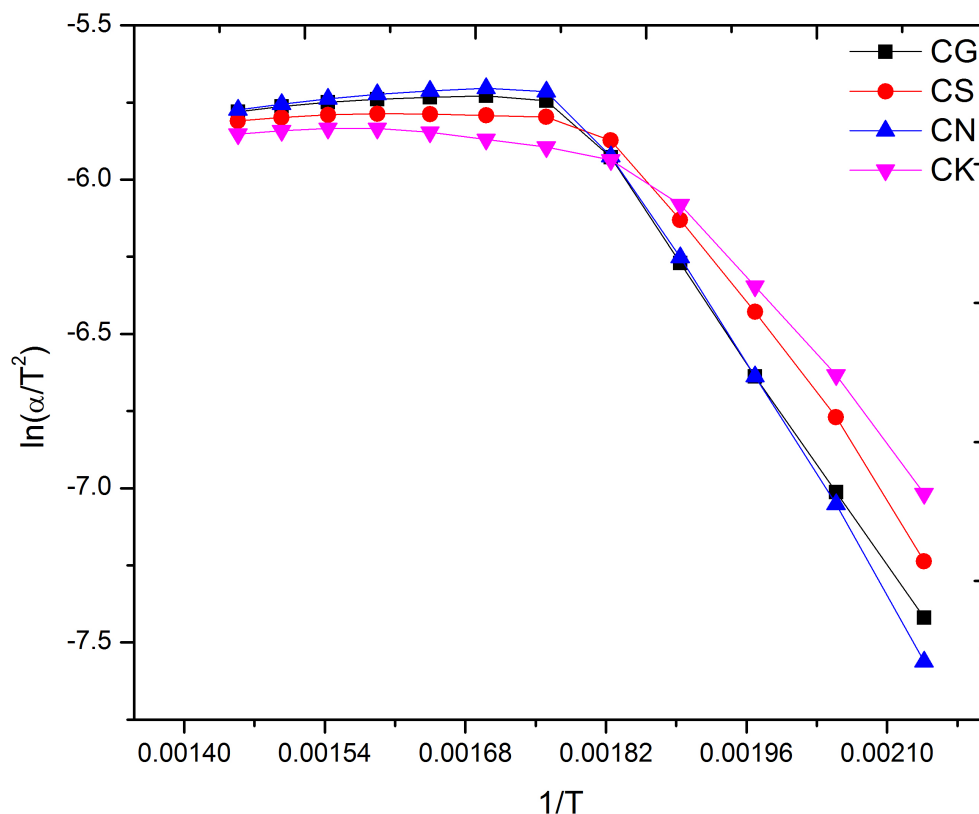


Fig. (9). Plot of Piloyan–Novikova equation $\ln(\alpha/T^2)$ v/s $1/T$ for copper surfactants derived from various oils.

By this equation, the values of energy of activation are observed to be in following order for CG, CS, CN and CK.

CG > CS (for edible oils)

CN > CK (for non-edible oils)

A perusal of Table 5 reveals that the value of activation energy is highest for the CN and smallest for the CS, irrespective of the equation applied signifying that saturated fatty acids require highest activation energy for decomposition. In general, CN derived from Neem oil showed higher thermal stability due to higher content of saturated fatty acids and MUFA. The higher PUFA content of CK from karanj oil makes it less stable and it requires lesser energy to degrade. Similarly, CG derived from Groundnut oil showed higher thermal stability due to higher content of saturated fatty acids and MUFA. The higher PUFA content of CS from Sesame oil makes it less stable and it requires lesser energy to degrade. The entropy of activation (ΔS), enthalpy of activation (ΔH) and free energy of activation (ΔG) were calculated using the following equation:

$$\Delta G = \Delta H - \Delta T \Delta S \quad (6)$$

$$\Delta H = E - RT \quad (7)$$

$$\Delta S = 2.303 \log \left(\frac{zh}{kt} \right) R \quad (8)$$

All copper surfactants molecules have negative entropy, which indicates that decomposition reactions proceed with lower rate than normal. The negative value of entropy also indicates that the activated complex has a more ordered and more rigid structure than the reactants or intermediates. The negative values of the entropies of activation are compensated by the values of enthalpies of activation, leading to almost the same values for the free energy of activation (Table 6).

Table 6. Thermodynamic parameters for the decomposition reaction of copper surfactants derived from various oils.

Compound	Method	Z (s ⁻¹)	ΔS^*	ΔH^*	ΔG^*
			(J K ⁻¹ mol ⁻¹)	(kJ mol ⁻¹)	(kJ mol ⁻¹)
CG	F.C.E.	1.19	-324.22	51.73	229.55
	C.R.E.	4.27	-237.55	49.83	180.21
	H.M.E.	17.12	-207.45	52.68	166.25
	B.E.	10.99	-230.34	59.79	185.87
	P.N.E.	0.142	-266.44	40.69	186.43
CS	F.C.E.	6.67 x 10 ⁻⁶	-309.71	43.72	213.43
	C.R.E.	6.98 x 10 ⁻⁴	-310.42	43.98	214.22
	H.M.E.	10.05	-230.8	32.34	158.83
	B.E.	3.911	-238.86	33.89	164.62
	P.N.E.	1.66 x 10 ⁻²	-284.85	31.82	187.45
CN	F.C.E.	3.16 x 10 ⁻⁷	-355.02	57.69	241.32
	C.R.E.	9.32 x 10 ⁻³	-288.82	56.31	214.62
	H.M.E.	63.81	-215.45	63.48	181.55
	B.E.	6.36	-234.62	66.27	194.84
	P.N.E.	0.300	-260.06	73.33	215.82
CK	F.C.E.	2.70 x 10 ⁻⁵	-300.73	38.42	202.96
	C.R.E.	2.92 x 10 ⁻⁴	-317.76	40.12	214.26
	H.M.E.	8.42	-232.31	38.93	166.24
	B.E.	3.08	-240.63	30.09	161.96
	P.N.E.	4.42 x 10 ⁻³	-295.03	24.98	186.62

CONCLUSION

Copper surfactants derived from edible and non-edible oils are found to be eco-friendly, completely biodegradable and nontoxic with significant antiviral, anti-cancerous, antifungal and antimicrobial properties. Based upon their widest applicability, copper surfactants were synthesized and characterized by H NMR, IR and ESR spectral analysis. TGA technique was done in order to determine energy of activation by applying equations like Freeman Carroll, Coats-Redfern, Horowitz-Metzger, Broido equation Piloyan –Novikova. This may be concluded by the above study that CS and CK soaps might be easily degradable naturally, biologically and thermally in comparison to CG and CN soaps. The products prepared by groundnut, sesame, neem and karanj should be promoted and encouraged due to their significance and degradability.

CONSENT FOR PUBLICATION

Not applicable.

CONFLICT OF INTEREST

The authors declare no conflict of interest, financial or otherwise.

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