



Characterization of Divalent Metal Soaps of *Cannabis Sativa* Seed Oil

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Highlights

- Metal soaps were prepared using oil from *Cannabis sativa*.
- Ba, Ca and Zn soaps were thermally stable as the major decomposition step occurred at 250 °C.
- Weight loss for the metal soaps were less than 10% at temperatures up to 200 °C.
- The activation energy had average values $> 4.0 < 11.0$ kJ/mol using the Broido equation.

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Abstract

In this study, the structural characteristics and thermal degradation behaviour of barium, calcium and zinc soaps of hemp seed oil prepared using the metathesis method are reported. Scanning electron micrographs of the metal soaps showed similar plate-like microstructures and are indicative of an orderly packed particulate structure. The bands at 1509, 1539 and 1543 cm^{-1} in the FTIR spectra showed the association of the carboxylate ion with Ba, Ca and Zn ions respectively. The thermal decomposition consisted of one single step on the weight loss – temperature curves located around 250 – 450 °C for all the divalent metal soaps at the various heating rates and the observed loss in weight which occurred before 200 °C was minimal (< 10%). The average values of the apparent activation energy for the thermal process were 18.73, 13.77 and 8.48 kJ/mol for Zn-HSO, Ca-HSO and Ba-HSO respectively.

1. INTRODUCTION

Additives from renewable resources continue to play a key role in the search for new materials with improved properties for potential applications: Vegetable oils are renewable resources which offer and continue to contribute significantly to new perspectives for sustainable development. Lots of studies abound in literature on the use of natural plant oil resources for industrial applications due to their abundant occurrence in all parts of the world, exceptional qualities of being non – toxic, biodegradable and environmentally friendly. These plant oils: soybean, jatropha, rubber, khaya, sunflower, linseed, etc., have been reportedly utilized as raw materials for the preparation of additives in polymer processing and in various other applications [1,2].

Soaps of alkaline – earth and transition metals with general formula $M(\text{C}_n\text{H}_{2n+1}\text{COO})_x$ where M is the metal ion and x is the charge, become essential ingredients of various industries: as heat and light stabilizers in plastics; driers in paintings; water-proofing agents on fabrics, etc., [3] and more recently as precursors of nanofilms, nanocomposites, nanoparticles and chemical thickeners in greases [4]. There are several reports in literature using metal carboxylates from natural plants such as linseed, soya bean, sunflower, rubber seed, for polymer processing; as thermal stabilizer and plasticizer [4-9], for example, metal soaps have been

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shown to substitute labile sites in polyvinylchloride (PVC) chains thereby stabilizing it against thermal degradation.

These metal soaps from vegetable oils which are renewable resources have continued to emerge as a subject of enormous scientific interest owing to their continuous use as additives and stabilizers in various industrial applications. Turkey is one of the world's richest countries in terms of plants for medicinal, aromatic as well as industrial uses [10]. *Canniba sativa L.* (hemp) is an angiosperm grown in temperate climates (humid atmosphere requiring an average monthly rainfall of at least 2.5 inches throughout the growing season) and generally harvest takes place in 17 weeks after attaining a height of about fifteen feet.

The two common compounds naturally found in hemp seed oil (HSO) are tetrahydrocannabinol and cannabidiol [11] having molecular formula $C_{21}H_{30}O_2$ (molecular mass = 314.469 g/mol). There are no previous reports on the application of the oil from *Cannabis sativa* (hemp) plant as aid in the processing of polymers.

The fatty acid profile and physicochemical properties of HSO are presented in Table 1.

Table 1. Fatty acid profile and physicochemical properties of HSO [11]

Parameter	Values
<i>Physicochemical properties</i>	
Colour	Bold Yellow
Acid value (mg KOH/g)	2.15
Iodine value (gI ₂ /100g)	163.5
Peroxide number (meq/kg)	7.2
Saponification value (mg KOH/g)	190.2
Refractive index (at 40°C)	1.4570
Specific gravity (at 20°C)	0.8927
<i>Fatty acid profile (wt.%)</i>	
Palmitic acid C16:0	5 – 9
Stearic acid C18:0	2 – 3
Oleic acid C18:1	10 – 16
Linoleic acid C18:2	50 – 70
Linolenic acid C18:3	15 – 40

This report is part of a continuous study aimed at the development of low-cost biobased materials which have found diverse industrial applications and examines the thermal behaviour of divalent metal soaps of HSO.

2. MATERIALS AND METHOD

HSO was obtained by cold-press (MP-001 Screw Press, Turkey) from hemp seed meal purchased from a local company (Özşen Lokman Hekim) in Turkey. The following metal salts: BaCl₂.2H₂O, CaCl₂. 2H₂O and ZnSO₄.7H₂O, (99%, Fluka, Steinheim, Germany) were used for the preparation of the metal soaps of HSO by metathesis as described previously [12]. The reaction involves treatment of the oil with 20% (w/v) sodium hydroxide in hot ethanol, then addition of 30% (w/v) aqueous solution of the appropriate divalent metal salt slowly and stirring the mixture adequately. The resulting metal soap formed was washed with warm water severally and recrystallized using petroleum ether. By using a Nüve EV 018 vacuum oven, the prepared metal soaps were dried under vacuum at room temperature conditions.

Scanning electron microscopy (SEM) (Quanta FEG 250) was used for to ascertain the morphology of the metal soap samples. The samples were coated with gold and palladium metals by using the sputtering technique. The structural composition of the metal soaps was evaluated by Fourier transform infrared (FTIR) spectroscopy using a DuraScope (Genesis II) spectrophotometer (Smiths Detection Inc., Danbury, CT, USA). FTIR spectra were recorded in the absorbance mode with a resolution of 1 cm⁻¹ in the range of 4000–400 cm⁻¹. The thermal degradation of the metal soaps of HSO was carried out on a thermogravimetric analyzer STA7300 (HITACHI) in the temperature range of 25 – 600 °C using three particular levels of

heating: 2, 5 and 10 °C min⁻¹ under nitrogen atmosphere to ascertain notable differences, if any, in the temperatures of extent of degradation at the various heating levels.

3. RESULTS AND DISCUSSION

3.1. Morphology and Particle Size of Metal Soaps of HSO

SEM measurements serve to visually identify the presence of metal soap agglomerates and the morphology of the divalent metal soaps prepared by metathesis is shown in Figure 1.

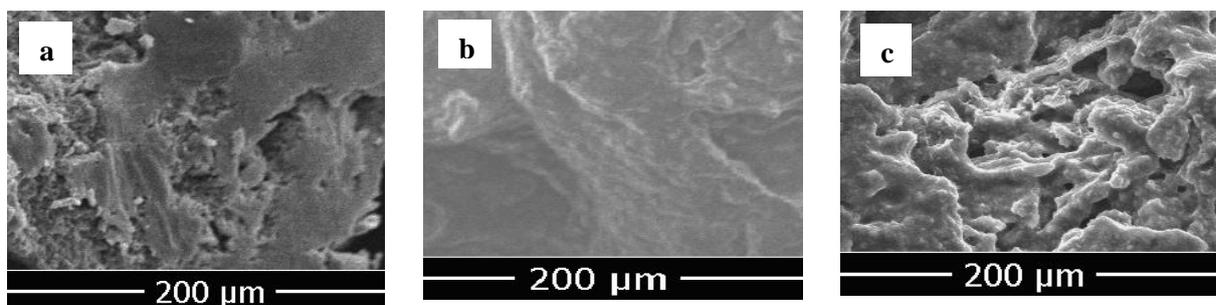


Figure 1. Micrographs of metal soaps of *Cannabis sativa* seed oil at x500 magnification a) Ba-HSO b) Ca-HSO c) Zn-HSO

Metal soaps of HSO show packed microstructures that is consistent with observed morphological structures for metal stearates [12] and metal soaps of rubber seed oil obtained in a previous study [7]. This is an indication of an orderly packed particulate structure with no obvious wide range within the particles in the system.

3.2. Structural Elucidation of Metal Soaps of HSO using FTIR

FTIR is a technique used in identifying the functional groups present in a material. The IR spectra of hemp seed oil and the divalent metal soaps (Ba, Ca and Zn) of HSO are shown in Figures 2 and 3, respectively. The spectra show the functional groups present in the samples, reactant conversion and impurities if present.

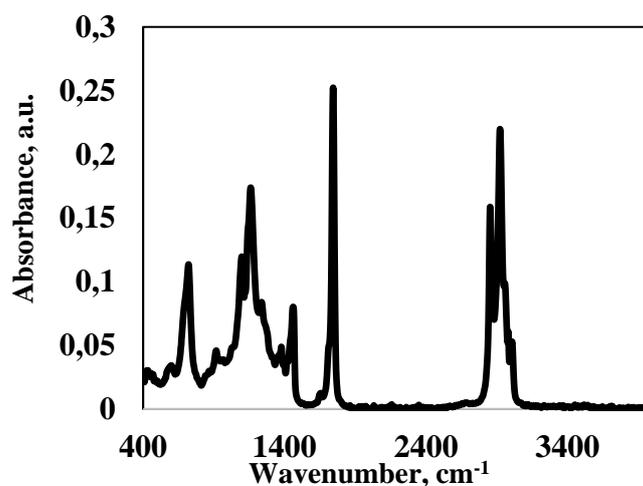
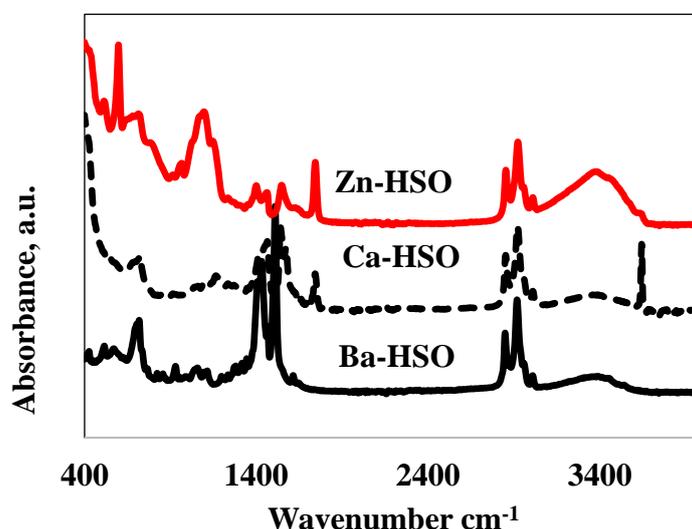


Figure 2. IR spectra of HSO

Table 2. Characteristic functional groups of HSO

Frequency (cm ⁻¹)	Functional group
2924	-C-H; stretching freq. of non-conjugated unsaturation
2852	-C-H; stretching freq. of an alkane
1750	-C=O; stretching freq. of an ester
1462	-CH ₂ ; bending freq. of unsaturated alkene
1164	-C-O; stretching freq. of an ester
721	-C-C; bending freq. of saturated carbon atom

The spectra of the metal soaps of HSO include the many common peaks expected from HSO shown in Table 2 as no modification occurs on the alkyl chain of triglycerides during saponification. The conversion of the oil to the soap is usually judged by the appearance and disappearance of some absorption peaks in the 1580 – 1550 and 1750 – 1700 cm⁻¹ region [13-15]. This means that the peak observed at 1750 cm⁻¹ due to C=O stretching vibration of the oil would disappear in Figure 2 while the characteristic band of the corresponding metal carboxylate between 1500 and 1540 cm⁻¹ due to COO⁻ stretching vibration would appear in Figure 3.

**Figure 3.** IR spectra of metal soaps of HSO

The absorption bands seen in Figure 3 appearing between 1745 and 1750 cm⁻¹ corresponding to C=O stretching vibration of free esters of HSO indicate the presence of unreacted oil in Ca-HSO (1743 cm⁻¹) and Zn-HSO (1743 cm⁻¹) but was absent in Ba-HSO. The characteristic band assigned to the asymmetric stretching mode for carboxylates were observed at 1509, 1539 and 1543 cm⁻¹ for Ba-HSO, Ca-HSO and Zn-HSO respectively and are similar to values reported in literature for metal stearates and carboxylates prepared from vegetable oils [3,7-8,12]. The bands appearing at 719, 721 and 713 for Ba, Ca and Zn soaps of HSO correspond to the bending vibration mode of the carboxylate [15].

Table 3. IR frequencies (cm⁻¹) and assignments for metal soaps of HSO

Ba-HSO	Ca-HSO	Zn-HSO	Band Assignment
3348	3644	3372	-OH stretching mode
2920	2924	2922	-CH ₂ asymmetric scissoring mode
2848	2850	2850	-CH ₂ symmetric scissoring mode
-	1743	1743	C=O stretching vibration
1509	1539	1543	COO ⁻ asymmetric stretching mode
1431	1462	1398, 1464	COO ⁻ symmetric stretching mode
719	721	713	COO ⁻ bending mode

The metal soaps exhibit the characteristic absorption bands of metal carboxylates as indicated in Table 3. The formation of soap has been reported [13, 15] to be marked by the disappearance of the very broad band attributed to the O-H stretch in the $3300 - 2500 \text{ cm}^{-1}$ region and the replacement of the bands assigned to the C=O and C-O around 1700 and 1300 cm^{-1} respectively with the bands attributed to the COO^- asymmetric and symmetric stretch around 1580 and 1400 cm^{-1} , respectively. The broad bands observed for Ba, Ca and Zn metal soaps of HSO centred at 3348 , 3644 and 3372 cm^{-1} respectively is the result of $-\text{OH}$ stretching modes that occurs about the region $3500 - 3200 \text{ cm}^{-1}$ and could be based upon the possible formation of the hydroxide of the metal especially for Zn-HSO which had the highest intensity [8].

During saponification, hydrolysis and precipitation occur simultaneously and unreacted metal ions, M^{2+} , react with excess hydroxide ions, OH^- , to form the metal hydroxide, $\text{M}(\text{OH})_2$, which cannot be removed by washing [7]. The bands appearing at 2920 , 2848 , 1431 for Ba-HSO, 2924 , 2850 , 1462 for Ca-HSO and 2922 , 2850 , 1464 , 1394 cm^{-1} for Zn-HSO correspond to $\nu_a\text{CH}_2$ (asymmetric), $\nu_s\text{CH}_2$ (symmetric) and $\delta_s\text{CH}_2$ (scissoring) modes due to the alkyl chain structure present in hemp seed oil. The bands appearing at 719 , 721 and 713 for Ba, Ca and Zn soaps of HSO correspond to the bending vibration mode of the carboxylate [15].

3.3. Thermogravimetric Analysis

An important feature used in evaluating the suitability of an additive for use as a stabilizer is its susceptibility to thermal degradation and ability to perform above the high temperatures needed for processing vinyl polymers. Typical TG thermograms obtained from the non-isothermal treatment of the divalent metal soaps of HSO from 25 to $600 \text{ }^\circ\text{C}$ at three different heating rates area is shown in Figure 4.

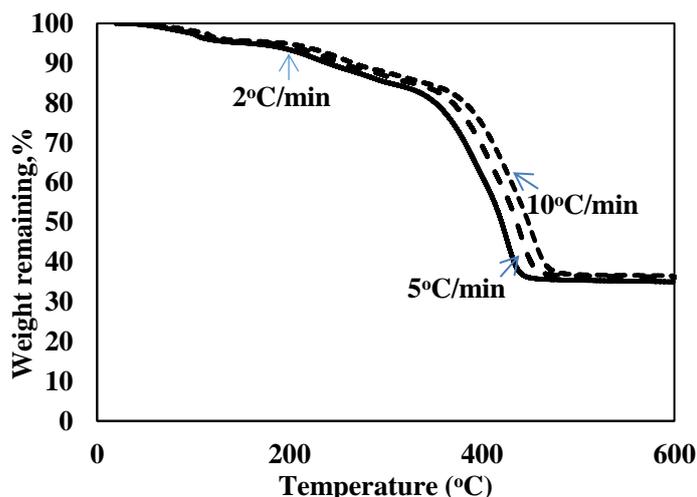


Figure 4. Thermograms of Zn-HSO at three different heating rates

From similar thermograms obtained for Ba-HSO and Ca-HSO, all the metal soaps exhibited an initial stable region up to 100°C with a first minimal phase transition occurring between $100 - 150^\circ\text{C}$ with a percentage weight loss of $< 5\%$ which could be attributed to the influence of the presence of traces of moisture in the prepared metal soaps and a second somewhat slow decomposition ($150 - 250^\circ\text{C}$) and a major decomposition which was rapid around $250 - 450^\circ\text{C}$ for all the divalent metal soaps at the various heating rates. The decomposition of metal carboxylates have been reported to be a multi-stage process with the nature and number of phase transitions being dependent upon the alkyl chain length and presence of unsaturation. During decomposition of metal carboxylates, three major decomposition steps occurring between $150 - 250^\circ\text{C}$, $250 - 350^\circ\text{C}$ and $350 - 450^\circ\text{C}$ ascertained with the decomposition of the unsaturated fatty acids such as linoleic acid, decomposition of monounsaturated fatty acids such as oleic acid and decomposition of the saturated fatty acids such as palmitic acid respectively have been identified as occurring during the thermal process [13,16].

The thermal stability of Ba, Ca and Zn soaps of HSO were calculated using different temperatures as these data provide information on the apparent stability of the material and the residual weight at 200°C and 600°C (Table 4). The loss in weight observed at temperatures up to 200°C had values $\leq 9\%$ for all the samples

attributed to moisture loss. The residual weight of the metal soaps samples at 200 °C was highest for Zn-HSO and would appear that it is more stable compared to Ba-HSO and Ca-HSO. The high residual content at this temperature may be attributed to the hydroxide present observed from the IR spectra which yields the metal oxide as part of the decomposition products. The products of the decomposition of metal carboxylates have been shown to be ketones as the main organic product, carbonates of the metal, the oxides and other gaseous products (Equation (1)) [13]



The apparent thermal stability observed for Zn-HSO compared with Ba-HSO and Ca-HSO may likely be connected to the fact that Zn^{2+} ions possess unsaturated d-orbitals, a common feature of transition metals, which probably confers some form of stability as the carboxylate anions form chelate bonds with the d-orbitals of the zinc ions.

Table 4. Data on thermal treatment of divalent metal soaps of HSO

Metal soap	Heating rate (°C/min)	Mass loss (°C)				Residual weight (%) at 200 °C
		5%	10%	30%	50%	
Ba-HSO	2	84.4	325.0	440.2	464.3	91.0
	5	98.2	348.5	454.7	478.6	91.5
	10	112.0	377.0	466.4	492.3	92.4
Ca-HSO	2	160.5	319.4	393.7	426.5	92.8
	5	156.0	339.6	412.0	443.1	93.2
	10	147.3	346.7	422.4	454.5	93.1
Zn-HSO	2	157.3	239.7	382.4	420.4	93.4
	5	179.6	253.8	398.4	436.1	94.3
	10	195.1	266.0	411.3	448.2	94.8

The observed difference in the temperatures of extent of degradation at the various heating rates is minimal, that is, there is no apparent relationship between the heating rate and change in mass during the decomposition process. It would seem from these results that variation of extent of degradation with heating rate is not significant. The temperatures to attain percentage conversions vary with heating rate and tended to be higher at higher rates of heating. The temperatures at which 5% mass loss occurred at 10 °C min⁻¹ heating rate for metal soaps of some selected vegetable oils such as *Hevea Brasilliensis* seed oil (RSO) [17], *Ximenia Americana* seed oil (XSO) [18] and *Balanites aegyptiaca* seed oil (BSO) [18] are given in Table 5.

Table 5. Relative thermal stabilities of metal soaps of BSO, RSO and XSO [17, 18]

Metal soap	Temperature at which 5% mass loss was attained (°C)
<i>Balanites aegyptiaca</i> seed oil (BSO)	
Ba-BSO	377.6
Ca-BSO	361.4
Zn-BSO	214.7
<i>Hevea Brasiliensis</i> seed oil (RSO)	
Ba-RSO	114.5
Ca-RSO	181.4
Zn-RSO	126.5
<i>Ximenia Americana</i> seed oil (XSO)	
Ba-XSO	340.2
Ca-XSO	324.1
Zn-XSO	156.0

From Table 4, the temperatures at which divalent metal soaps of HSO attained 5% conversion using 10 °C min⁻¹ heating rate compare favourably with metal soaps of RSO though lower when compared with metal soaps of BSO and XSO. These vegetable oils are polyunsaturated triglycerides containing oleic, linoleic

and linolenic fatty acids in various proportions and the observed differences may be attributed to the level of unsaturation present in the fatty acid moiety of the oils.

In our previous studies, the single-run method of Broido have been used for quick estimation of the kinetic parameters for the entire decomposition process of metal soaps prepared from vegetable oils at a single heating rate [19]. The Broido method is a simple integral method using a single curve

$$\ln\{\ln(1/\alpha)\} = -\frac{E}{RT} + \ln\left(\frac{RZT_m^2}{\beta E}\right). \quad (2)$$

The Broido's equation (Equation 2), allows the value of E to be obtained from the slope of the linear plot of $\ln(\ln 1/\alpha)$ vs $1/T$. The equation have been derived on the basis of the assumption that the rate of decomposition is a function of two variables, temperature (T) and fractional decomposition (α) at constant pressure. The dependence of the rate of the decomposition process on temperature represented by the rate constant $k(T)$ and the dependence on the extent of conversion by the reaction model, $f(\alpha)$ is expressed as Equation 3 and describes the rate of a single step process

$$\frac{d\alpha}{dt} = k(T) f(\alpha). \quad (3)$$

The extent of conversion is determined experimentally as a fraction of the overall change in a physical property (weight) accompanying the decomposition process $\left[\alpha = \frac{W_r - W_\infty}{W_o - W_\infty} \right]$.

In this study, Broido plots were drawn using temperatures falling within the range 150 – 450 °C, at which various conversions (5, 10, 15 and 20%) were obtained to represent the average value of the apparent activation energy of the decomposition process. The processing of vinyl polymers, for example, PVC, falls within this temperature range (190 – 220 °C) and thermal stabilizers which are added prior to processing should be stable to thermal treatment at this temperature.

Thermal stabilizers are additives used in the processing of polymers, for example, PVC, because of the poor thermal stability of the polymer and the use of different thermal stabilizers such as metal soaps; epoxy compounds amongst others have been studied extensively over the past 20 years. The average value of the apparent activation energy determined within certain degrees of conversion (α) have been recommended because solid-state reactions have been reported to be unstable at the beginning and ending of the thermal decomposition process [20]. The plots for the kinetic rate data at a fixed heating rate (β) obtained for the non – isothermal decomposition of the divalent metal soaps of HSO at the different heating levels (2, 5 and 10 °C/min) using the linear relationship of $\ln(\ln 1/\alpha)$ versus $1/T$ are shown in Figure 5 using Zn-HSO as a typical example.

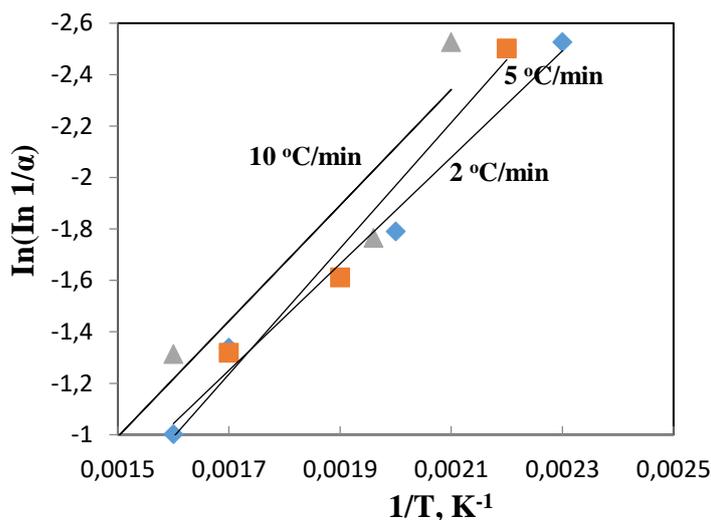


Figure 5. Broido plots for Zn-HSO

The average values of the apparent activation energy obtained for the thermal decomposition of metal soaps of HSO were 18.73 kJ/mol for Zn-HSO, 13.77 kJ/mol for Ca-HSO and 8.48 kJ/mol for Ba-HSO. The E_a of decomposition was highest for Zn-HSO which suggests that it is the least prone to degrade and somewhat agree with the results observed in Table 4 on the residual weights obtained at 200 °C. Thus, if values of E_a are used as estimates of thermal stability, these results would suggest that Zn-HSO would confer the highest stabilizing effect in the thermal degradation of PVC. These values correspond fairly well with the values of 19.55 kJ/mol and 13.46 kJ/mol obtained for barium and calcium metal soaps of rubber seed oil (RSO) respectively at a heating rate of 10 °C min⁻¹ [1].

4. CONCLUSION

In this study, the structural characteristics and thermal behaviour of barium, calcium and zinc soaps of hemp seed oil were conducted. The metal soaps from the SEM micrographs exhibited plate-like microstructures in the lamellar form while the bands at 1509, 1539 and 1543 cm⁻¹ in the FTIR spectra showed the association of the carboxylate ion with Ba, Ca and Zn ions respectively. It has been observed that the metal soaps thermally stable up to an approximate value of 250 °C as the weight loss at this temperature range was less than 10% and is considered an excellent guide before its use as additives in a myriad of industrial applications. The average values necessary for the thermal decomposition process were 18.73, 13.77 and 8.48 kJ/mol for Zn-HSO, Ca-HSO and Ba-HSO respectively. The results show that the thermal stability of metal soaps of HSO compare favourably with metal carboxylates of some vegetable oils many of which are used as commercial heat stabilizing additives in the processing of PVC.

CONFLICTS OF INTEREST

No conflict of interest was declared by the authors.

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