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Research Article
Physicochemical and Kinetics Parameters of *Allanblackia floribunda* Seed Oil and some of its Metal Carboxylates

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Abstract
Background: Nigerian flora is rich in oilseeds that have not been fully explored for industrial applications. Many of the seed oils have been characterized but are yet to be explored maximally for the production of chemical intermediates and other useful products. One of such seed oils is *Allanblackia floribunda* seed oil (ASO). There is no record of utilization of ASO for the preparation of metal carboxylates in the literature, hence we decided to embark on this study. Metal carboxylates have many industrial applications including thermal stabilizers of poly(vinyl chloride). The saturated nature of the seed oil makes it a suitable feedstock for the preparation of metal carboxylates. **Materials and Methods:** The preparation of barium, calcium, magnesium and zinc carboxylates of the seed oil was carried out by precipitation method involving metathesis in ethanol. Evidence for the formation of the carboxylates was derived from FTIR spectrophotometry measurement. Thermal stability study on the carboxylates was carried out gravimetrically in the temperature range 160-200°C and kinetics parameters of their decomposition calculated. **Results:** The carboxylates showed two asymmetric vibrations and two symmetric vibrations of the carboxylate group in the spectra. The two asymmetric vibrations occurred in the range 1633-1512 cm^{-1} while the two symmetric vibrations were observed in the range 1471-1398 cm^{-1} suggesting the binding mode of the carboxylate groups to the metal ions and confirmed formation of the carboxylates. The values of rate constant for the decomposition of the metal carboxylates are of the order 10^{-3} min^{-1} and increased with temperature. The values of E_a ranged from 11.7-21.5 kJ mol^{-1} while $\Delta H^{\ddagger}$ ranged from 7.9-17.7 kJ mol^{-1} and the process is endothermic. The values of $\Delta G^{\ddagger}$ (131.4-132.8 kJ mol^{-1}) and $-\Delta S^{\ddagger}$ (0.252-0.273 kJ mol^{-1} K^{-1}) indicate that the decomposition of the metal carboxylates is a non-spontaneous process and the result of free energy of activation showed Zn-ASO as the most stable. **Conclusion:** The results revealed that *Allanblackia floribunda* seed oil could serve as feedstock for the preparation of metal carboxylates and this will add commercial value to it thereby encouraging its cultivation in Nigeria.

Key words: Thermal stability, kinetics, decomposition, seed oil, metal carboxylates, unsaturation

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Data Availability: All relevant data are within the paper and its supporting information files.

INTRODUCTION

Seed oils are important renewable resource that have both nutritional and industrial applications. There has been renewed interest in the substitution of petroleum feedstock with seed oils in many industrial applications based on the depletion of the petroleum feedstock, a non-renewable resource and environmental concern[1]. Many of the materials arising from the seed oils have competitive values in terms of cost and performance when compared with petroleum based products. They have found application in the production of paints, inks, cosmetics, pharmaceuticals and chemical intermediates such as metal carboxylates[2]. This is possible due to the fact that seed oils contain triglycerides with suitable functional groups such as ester, alcohol, triple and double bonds either conjugated or non-conjugated and other functional groups in their molecule that can undergo several chemical modifications. This fact is responsible for their ability to replace petroleum feedstock in many applications.

There are vast varieties of widely grown plants and herbs in Nigeria and the Nigerian flora has been described as one of the most extensive floras in the African continent[3]. The seeds of many of these plants contain a large amount of oils, which are viable alternative of petroleum resources for many applications. Many of the oils have been characterized and explored in the preparation of many chemical intermediates such as alkyd resins, metal carboxylates, biodiesel etc[1,4-7] however, so many are yet to be fully explored as industrial raw materials. One of such is *Allanblackia floribunda* seed oil, also known as tallow tree seed oil.

Allanblackia floribunda belongs to the family Guttiferae and genus *Allanblackia*, it is known as izeni and uzoka (Edo), orogbo-erin (Yoruba), egba (Ibo), ediang (Efik), obobio-obo (Ijaw).

It is an evergreen medium-sized tree up to 30 m tall with a cylindrical or slightly fluted trunk and a narrow crown supporting horizontal branches. It has a fairly smooth bark and simple opposite leathery leaves evenly distributed along the branches of the tree[8]. A matured tree produces about 100-150 egg-shaped brown fruits of weight between 1.2 and 4.0 kg. *A. floribunda* grows in parts of Central, East and West African countries including Nigeria, Ghana, Democratic Republic of Congo, Uganda and Tanzania where it grows naturally in moist lowland and upland rainforest zones[9].

The fruits contain 25-40 oil-rich brown seeds, with a seed containing 68-72% oil[10,11]. *Allanblackia floribunda* seed oil (ASO) contains mostly of stearic and oleic acids which have always been part of human diet and also reported to lower plasma cholesterol levels, thus reducing the risks of heart attack hence, *A. floribunda* seeds are used for treating hypertension[12]. The presence of stearic acid as a constituent of its fatty acid is a great potential for its application in pharmaceutical preparations.

The seed oil of *A. floribunda* has been well characterized and information available in the literature revealed that it is a non-drying oil. However, there is no record of its utilization in the preparation of metal carboxylates. An investigation of the possible usage of the seed oil in preparing metal carboxylates and characterising them will be a worthwhile exercise. Also, the competition between the industrial requirement for oil and their use for edible purposes has made it imperative to find alternative sources of oils from unexploited and underutilized plants which can serve both industrial and domestic purposes. The aim of this study is to prepare some metal carboxylates of *A. floribunda* seed oil, characterize them so as to add value to this unpopular oil in Nigeria.

MATERIALS AND METHODS

Determination of physicochemical properties: The *Allanblackia floribunda* oilseeds collected in the University farm at Okitipupa, Ondo State were sun-dried, deshelled, pulverised and extracted with n-hexane using soxhlet extraction method. These were determined using standard AOCS methods[13].

Preparation of metal carboxylates: The carboxylates were prepared as earlier reported[5] and dried in an oven at 60°C for 24 h. Analar grade of the salts were used for the preparation.

FTIR spectroscopy of the carboxylates: The FTIR spectra of the oil and metal carboxylates were measured using a SHIMADZU FTIR-8400S at a scan rate of 4 cm^{-1} between wave numbers 4000-400 cm^{-1}.

Thermal stability studies on the metal carboxylates: The thermal stability of the carboxylates was investigated gravimetrically at 160, 180 and 200°C as a function of time by heating in muffle furnace. Typically, 3 g of the metal carboxylate was accurately weighed into a previously weighed crucible and heated in the muffle furnace at the appropriate temperature for a definite period of time (30-120 min). At the end of the heating time, the sample was withdrawn from the furnace and cooled in a desiccator. The residual weight of the sample was measured and the weight loss determined using the expression:

$$\text{Weight loss (\%)} = \frac{w_o - w_1}{w_o}$$

RESULTS

The fruit of *Allanblackia floribunda* is shown in Fig. 1 while some of the physicochemical characteristics of *Allanblackia floribunda* seed oil are presented in Table 1. Saponification value of the oil (185.7) showed that it is rich in long chain fatty acids. The peroxide value of 3.3 and the acid value of 0.6 revealed that the oil is stable against rancidity and oxidation. The oil content of the seed is 61%. The FTIR spectrum of the seed oil is presented in Fig. 2 while that of its barium carboxylate, Ba-ASO is shown in Fig. 3 representing those of other carboxylates based on similarity. The FTIR bands of *A. floribunda* seed oil and those of the metal carboxylates are presented in Table 2. The seed oil spectrum is characterized by band at 1735 cm^{-1} that is attributed the C=O stretching vibration of the triglyceride (oil). The metal carboxylates are characterized by two bands in the range 1633-1512 and 1471-1398 cm^{-1}. The former is ascribed to asymmetric stretching vibration of the carboxylate group while the latter is attributed to symmetric stretching vibration of the carboxylate group. The rate constants for the decomposition of metal carboxylates of the oil are presented in Table 3 and the result shows the dependence of rate constant on temperature. The values increased with temperature. The average rate constants are 5.8×10^{-3}, 5.4×10^{-3}, 6.6×10^{-3} and 4.6×10^{-3} min^{-1} for Ba-ASO, Ca-ASO, Mg-ASO and Zn-ASO, respectively. These values were used to calculate other kinetics parameters at the average temperature of 180°C. Other kinetics parameters for the decomposition process (E_a, $\Delta H^{\ddagger}$, $\Delta G^{\ddagger}$ and $\Delta S^{\ddagger}$) are presented in Table 4.

Fig. 1: Fruit of *Allanblackia floribunda*

Table 1: Physicochemical characteristics of *A. floribunda* seed oil

Characteristics	Value
Oil content (%)	61.2
Saponification value (mg KOH g^{-1})	185.7
Acid value (mg KOH g^{-1})	0.6
Peroxide value (mEq kg^{-1})	3.3
State at ambient temperature	Solid
Colour	Light yellow

DISCUSSION

The oil content of the seed is 61%, this is in agreement with the value of 60-66% reported in the literature[14]. Literature survey showed that fatty acid composition of the oil is mainly made up of a saturated fatty acid (stearic acid, 57%) and a monounsaturated acid (oleic acid, 39%) with trace amount of another saturated fatty acid (palmitic acid, 3%)[14]. The preponderance of saturated fatty acids in the oil showed that it is a non drying oil and occurred as light yellow solid at ambient temperature when the extraction solvent was removed. Iodine values in the range 35-39 g I_2/100 g had also been reported for the oil[11,14], further confirming the oil as

Table 2: FTIR bands for *A. floribunda* seed oil and the metal carboxylates

Assignment	ASO	Ba-ASO	Ca-ASO	Mg-ASO	Zn-ASO
O-H stretch	3443w	3458w	3419vs	3412vs	3510s
CH_2, C-H asymmetric stretch	2918vs	2918vs	2918vs	2918vs	2918vs
CH_2, C-H symmetric stretch	2850vs	2848vs	2850vs	2850vs	2848vs
C=O stretch	1735vs	-	-	1747w	-
COO^- asymmetric stretch	-	1556w, 1512vs	1575s, 1541s	1633s, 1573s	1618w, 1541vs
COO^- symmetric stretch	-	1469s, 1442s	1471s, 1435s	1467s	1465s, 1398s
C-O stretch+O-H in plane deform	1471m	-	-	-	-
C-O stretch	1255w	-	-	-	-
OH out of plane deform	-	931w	-	-	-
CH_2 rocking	717w	719w	721w	721w	744w, 721w

vs: Very strong, s: Strong, w: Weak, m: Medium

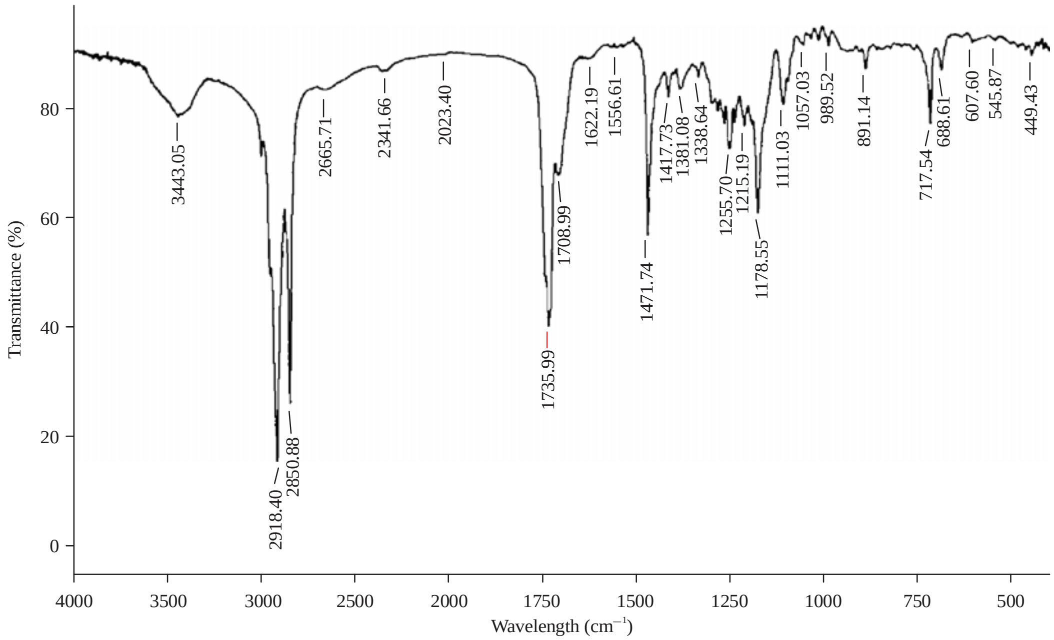

Fig. 2: FTIR spectrum of ASO

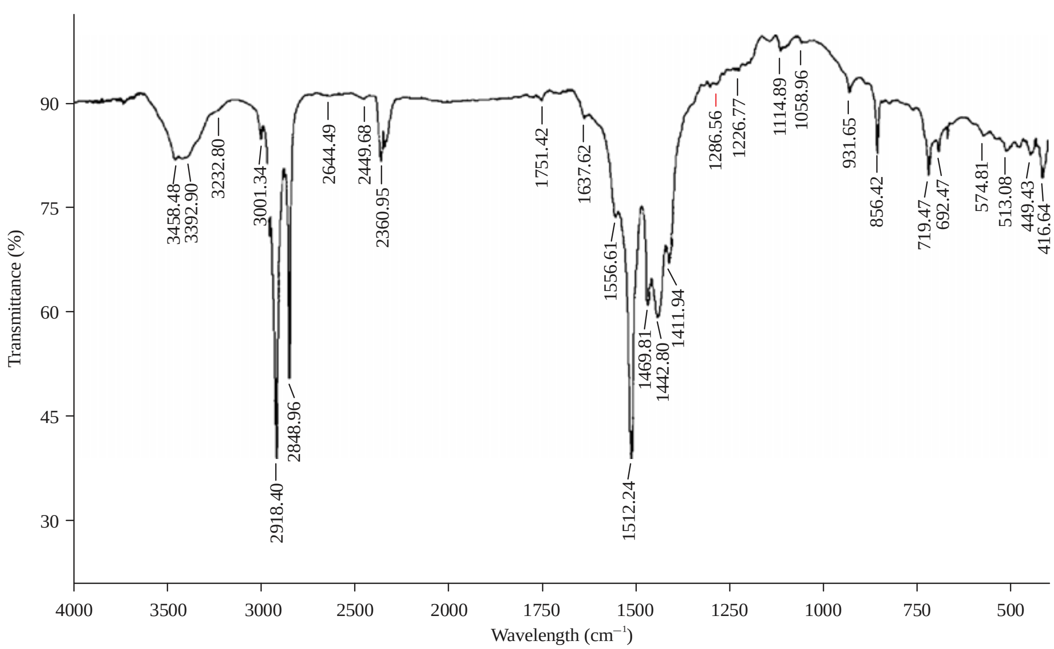

Fig. 3: FTIR spectrum of Ba-ASO

non-drying. The saponification value of 185.7 for the oil (Table 1) is comparable with 199 and 200 earlier reported and indicates the richness of the oil in high molecular weight fatty acids. Coconut oil is rich in low-molecular weight fatty acids and has high saponification value (251-264)[15]. The lower the saponification value, the higher the content of high-molecular weight fatty acids and vice-versa. The low peroxide value of 3.3 and acid value of 0.6 indicate the freshness of the oil and its stability against oxidation and rancidity.

The spectrum of seed oil is characterised by a strong band at 1735 cm^{-1} which could be attributed to ν(C=O) of an ester or free acid present in the oil. The band in the range 1300-1100 cm^{-1} is also attributable to ν(C-O) of the triglyceride of the oil. The CH_2 asymmetric and symmetric stretching vibrations occur with very strong intensity at

Table 3: Rate constants for the decomposition of metal carboxylates of *A. floribunda* seed oil

Metal carboxylate	Temperature (°C)	Rate constant ($k\times10^3$ min^{-1})	Average rate constant ($k\times10^3$ min^{-1})
Ba-ASO	160	4.1	5.8
	180	6.5	
	200	6.8	
Ca-ASO	160	4.4	5.4
	180	5.4	
	200	6.5	
Mg-ASO	160	5.7	6.6
	180	6.7	
	200	7.5	
Zn-ASO	160	4.0	4.6
	180	4.0	
	200	6.0	

Table 4: Thermodynamic parameters for the decomposition of metal carboxylates of *A. floribunda* seed oil

Metal carboxylate	E_a (kJ mol^{-1})	$\Delta H^‡$ (kJ mol^{-1})	$\Delta G^‡$ (kJ mol^{-1})	$\Delta S^‡$ (kJ mol^{-1} K^{-1})
Ba-ASO	21.5	17.7	131.9	-0.252
Ca-ASO	16.6	12.8	131.7	-0.262
Mg-ASO	11.7	7.9	131.4	-0.273
Zn-ASO	17.3	13.5	132.8	-0.263

ASO: *Allanblackia floribunda* seed oil, FTIR: Fourier transform infrared, Ba-ASO: Barium carboxylate of *Allanblackia floribunda* seed oil, Ca-ASO: Calcium carboxylate of *Allanblackia floribunda* seed oil, Mg-ASO: Magnesium carboxylate of *Allanblackia floribunda* seed oil, Zn-ASO: Zinc carboxylate of *Allanblackia floribunda* seed oil

2918 and 2850 cm^{-1}, respectively. The weak but broad band at 3443 cm^{-1} is assigned to v(O-H) of free acid in the oil. Except the bands at 2918 and 2850 cm^{-1} in the spectrum of the oil, all other bands disappeared in the spectra of corresponding metal carboxylates. Complete disappearance of the carbonyl band near 1700 cm^{-1} in the spectra of the carboxylates is an indication of a complete resonance between the two C=O bond of the carboxylic groups of the carboxylates[16].

Salts of carboxylic acids do not show a carbonyl band, rather, strong bands due to the asymmetric and symmetric stretching vibrations of the carboxylate group are observed at 1650-1550 and 1420-1300 cm^{-1}. The four spectra of the metal carboxylates showed characteristic two asymmetric and two symmetric carboxylate vibrations indicating their binding mode as bridging bidentate. For Ba-ASO, the two asymmetric vibrations occurred at 1556 and 1512 cm^{-1} while the two symmetric vibrations were observed at 1469 and 1442 cm^{-1}. The asymmetric vibrations of the carboxylate group of Ca-ASO were observed at 1575 and 1541 cm^{-1} and the two symmetric vibrations occurred at 1471 and 1435 cm^{-1}. For Mg-ASO, the asymmetric vibrations of the carboxylate group were observed at 1633 and 1573 cm^{-1} and the symmetric vibration occurred at 1467 cm^{-1}. In the case of Zn-ASO, the asymmetric vibrations were observed at 1618 and 1541 cm^{-1} with symmetric vibrations appearing at 1465 and 1398 cm^{-1}. This is consistent with reports on some carboxylates[5,17-19], thereby confirming the formation of the metal carboxylates. The difference in asymmetric and symmetric carboxylate stretching vibrations, $\Delta v = v_{asym}\text{-}v_{sym}$ has been used as an empirical indicator of their coordination mode. A difference, $\Delta v>200$ cm^{-1}, indicates a monodendate coordination mode[20]. For the carboxylates, Δv values ranged from 114-210 cm^{-1} with Ba-ASO having the least value of 114 cm^{-1} and Zn-ASO having the highest value of 210 cm^{-1}. The Ca-ASO and Mg-ASO have values of 140 and 166 cm^{-1}, respectively. Similarity in the absorption bands of the oil and the metal carboxylates was observed in the stretching vibrations of CH_2 groups of the alkyl group of fatty acid moiety of the carboxylates. The CH_2 asymmetric and symmetric stretching vibrations of the alkyl group occurred in the range 2918-2848 cm^{-1} for all the carboxylates. The fact that the stretching vibrations of the CH_2 groups remained unchanged indicates that they are not involved in the formation of the carboxylates. All the carboxylates are characterized by broad bands in the range 3510-3412 cm^{-1}, this could be attributed to hydrogen bonded OH groups possibly from water.

Thermal stability of the carboxylates was investigated gravimetrically by heating in muffle furnace at 160, 180 and 200°C within regular time intervals of 30-120 min.

The thermal decomposition rate of metal carboxylates follows a first order kinetics[21,22] and can be expressed as follows in Eq. 1:

$$\frac{dw}{dt} = k(w_o - w_1) \tag{1}$$

where, w_0 is the initial weight of the metal carboxylate and w_1 is the residual weight of the metal carboxylate after heating, with k as the rate constant. Rearrangement and integration of Eq. 1 gave Eq. 2:

$$\log(w_o - w_1) = \log w_o - \frac{kt}{2.303} \tag{2}$$

From the plots of logarithm of percentage weight loss against time, the values of the rate constant for the decomposition of the metal carboxylates were obtained and the result presented in Table 3. Rate constant is an indication of energy barrier a reaction needs to overcome and there exists an inverse relationship between the two parameters[23]. The smaller the barrier, the greater the rate constant. The calculated rate constants are temperature-dependent and of the order 10^{-3} min^{-1} with increase of 65.9, 47.7, 31.6 and 50.0% observed within the temperature range for Ba-ASO, Ca-ASO, Mg-ASO and Zn-ASO, respectively. Based on the dependence of rate constant on temperature, values for the activation energy (E_a) of the decomposition of the metal carboxylates were calculated using the relationship as in Eq. 3:

$$\log\frac{k_2}{k_1}=\frac{E_a}{2.303R}\left(\frac{T_2-T_1}{T_2T_1}\right) \quad (3)$$

and the result presented in Table 4.

Activation energy for the decomposition process range from 11.7-21.5 kJ mol^{-1} with Mg-ASO having the least value of 11.7 kJ mol^{-1} while Ba-ASO has the highest value of 21.5 kJ mol $^{-1}$. The Ca-ASO has a value of 16.6 kJ mol^{-1} and that of Zn-ASO is 17.3 kJ mol^{-1}. The values are consistent with figures earlier reported for some metal carboxylates[22,24,25]. The enthalpy of activation of the decomposition process, $\Delta H^{\ddagger}$ was calculated using the relationship: $\Delta H^{\ddagger} = E_a$-RT and the result presented in Table 4. From the values of $\Delta H^{\ddagger}$, the process was observed to be endothermic. Actual energy barrier to a reaction is usually indicated by free energy of activation[23] and this was calculated using the relationship as in Eq. 4:

$$\Delta G^{\ddagger}=-RT\left(2.303\log\frac{kh}{Tk_B}\right) \quad (4)$$

Most reactions are driven by a change in both enthalpy and entropy, some by a change in enthalpy while some others by a change in entropy[23], the entropy of activation, $\Delta S^{\ddagger}$ for the decomposition of the metal carboxylates was calculated using the expression as in Eq. 5:

$$\Delta S^{\ddagger}=\frac{\left(\Delta H^{\ddagger}-\Delta G^{\ddagger}\right)}{T} \quad (5)$$

The calculated values $\Delta G^{\ddagger}$ and $\Delta S^{\ddagger}$ are also presented in Table 4. The values obtained for $\Delta G^{\ddagger}$ and $\Delta S^{\ddagger}$ show that the decomposition of the metal carboxylates is a non-spontaneous process and the result of free energy of activation showed Zn-ASO as the most stable. Thermal stability of metal carboxylates prepared from seed oils depends on factors which include nature and length of carbon chain of the fatty acids, the metal ion involved as well as the level of unsaturation of the oil[24]. High level of unsaturation in the fatty acid moiety of the oil has inverse effect on thermal stability of metal carboxylates. The results of this study are compatible with the results reported early[2,21,22,24] which showed that metal carboxylates could be prepared from seed oils and are thermally stable at temperature up to 250°C.

CONCLUSION

The suitability of *Allanblackia floribunda* seed oil being a polysaturated oil was investigated for the preparation of metal carboxylates. Four metal carboxylates of the oil, Ba-ASO, Ca-ASO, Mg-ASO and Zn-ASO were prepared by precipitation via metathesis in ethanol. The FTIR spectrophotometry was used to confirm the formation of the carboxylates. Kinetics study of the decomposition of the carboxylates, carried out within 160-200°C range of temperature, showed that the decomposition followed first order kinetics and the process is endothermic and non spontaneous. The activation energies for the decomposition of the metal carboxylates indicate potential industrial applications. It is possible to develop PVC thermal stabilizers from this oil.

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