



Research Article

Synthesis and Characterization of New Polymers from *Adenopus breviflorus* benth oil, Styrene and Divinyl benzene by Cationic Polymerization

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Abstract

A variety of new polymers ranging from soft to hard plastics were prepared by cationic polymerization of *Adenopus breviflorus* oil with styrene and divinyl benzene initiated by modified boron trifluoride etherate. The fully cured thermosets were found to contain between 78- 92% cross linked materials possessing crosslink densities of between 2.39×10^3 to 2.09×10^4 mol/m³ and glass transition temperatures of 12 – 80°C. The tensile moduli of the materials ranged from 1.47 to 404MPa, the ultimate tensile stress varied from 0.18 to 7.78MPa and the elongation at break varied between 2 and 34%. Depending on composition, some of the materials possessed good damping and shape memory properties. Overall the newly prepared materials from *Adenopus breviflorus* oil hold a lot of promise as new polymeric materials.

Keywords: *Adenopus breviflorus* seed oil; cationic polymerization; thermoset properties

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Introduction

The use of renewable resources derived from plants is increasingly becoming more attractive due to its potential of meeting a number of objectives, including more diverse markets for farmers, reduced use of fossil fuels and reduced volumes of wastes going into landfills [1]. In particular, plant oils which are found in many parts of the world and are easily replenished have been reported to have the potential to replace petroleum as a major chemical feedstock for making polymers [2]. For example Li et al. [3-5] have reported the preparation of polymers ranging from rubbers to hard plastics by cationic polymerization of variety of oils: fish, tung and soybean oil with petroleum based co-monomers such as styrene, divinyl benzene and dicyclopentadiene in the presence of boron trifluoride etherate as initiator. Some other researchers, [6-8] have tried developing rigid resins from soybean oil.

Adenopus breviflorus (ADB) is grown in the middle belt area of Nigeria and at maturity it produces seeds which are mostly utilized as a soup ingredient in the Mid – Western part of Nigeria. Akintayo and Bayer [9] have reported that the seeds have an oil yield of 56.2% and the oil has a fatty acid composition comprising of palmitic acid (10.8%), stearic acid (14.1%); oleic acid (13.8%) and linoleic acid (61.3%). Based on this fatty acid composition, *Adenopus breviflorus* oil (ADBO) has a triglyceride structure with approximately 4.1 C=C double bonds per molecule on average in the fatty acid chain. This level of un-saturation is same as that obtainable for corn oil [10] which has successfully been used to prepare rigid thermosetting resin. Therefore in line with Nigerian government initiative to encourage industrial development based on its natural resources endowment, this study seeks to synthesize different polymeric materials by the cationic copolymerization of ADBO with styrene and divinylbenzene initiated by modified boron trifluoride etherate as catalyst. The structure, thermophysical, thermal and mechanical properties of the resulting polymers are discussed.

Experimental

Materials

Adenopus breviflorus (ADB) seeds were purchased from Erekesan market in Ado-Ekiti, Ekiti State Nigeria. The good seeds were washed with water and air dried and later milled using Christy mill. Oven dried samples (103⁰C) were extracted by Soxhlet method using n- hexane. Solvent was removed under reduced pressure on a rotary evaporator. The crude oil was refined by agitating with 18M NaOH (1:30g/g of alkali: powder) for 15min. The resultant mixture was heated to 75-80⁰C to break the soap stock and the neutral oil separated by centrifugation. Styrene (ST) and divinylbenzene (DVB) were purchased from Aldrich Chemical Company and used as received. Distilled grade boron trifluoride etherate (BF₃E) also from Aldrich chemical (Milwaukee, WI) was modified with *Adenopus breviflorus* oil (ADBO) ester [referred to in this work as modified catalyst (MC)] before being used as initiator in the reaction.

Cationic polymerization and nomenclature

The desired amounts of ST and DVB were added to the desired amount of ADBO. The reaction mixture was vigorously stirred, followed by the addition of an appropriate amount of a modified initiator which was generally required to produce homogeneous resins and polymers. The modified initiator was prepared by mixing ADBO ethyl ester with BF₃E. The reaction mixture was then injected into a Teflon mold and cured as follows: 60⁰C for 10 minutes, 80⁰C for 20 minutes and then 110⁰C for 24 hours. The nomenclature adopted in the study for the polymer samples was based on the original composition of the reactants. For example ADBO30–ST46–DVB16–MC8 represent a polymer prepared from 30 weight % ADBO, 46 weight% ST, 16 weight % DVB and 8 weight % modified initiator (5 wt % ADBO ester +3 wt% BF₃E).

To study the effect of composition of polymer matrix, a series of polymer was prepared where the oil and initiator content were held constant but the concentrations of ST and DVB varied while keeping constant the total percentage of the ST-DVB co-monomer.

Polymer characterization

Soxhlet Extraction

Bulk polymer sample of known weight was extracted with refluxing methylene chloride over a period of 24hr in a Soxhlet extractor. The resulting solution was concentrated by rotary evaporator and subsequent vacuum drying and the soluble substances characterized by ¹HNMR spectroscopy.

¹HNMR spectroscopic characterization

The ¹HNMR spectra of the extracted soluble substances were recorded in deuteriated chloroform using Avance 400MHz (Bruker, Germany)

Dynamic Mechanical Analysis (DMA)

The dynamic mechanical properties of the polymers were obtained on a TA Instrument dynamic mechanical analyzer, Q800 (TA instruments, New Castle, DE, USA) in the three point bending mode with a static force of 100MPa and dynamic force of 110mN. Thin sheet specimens 2mm in thickness and 5mm in depth were used and the spin- to –depth ratio was maintained at approximately 2. Each specimen was first cooled to – 60⁰C and then heated at 3⁰C /minute and a frequency of 1Hz under nitrogen. The visco-elastic properties viz; the storage modulus (E') and mechanical loss tangent (tan δ damping) or loss factor were recorded as a function of temperature. The glass- transition temperature (Tg) of the polymers were obtained from the peak of the tan δ curve. The damping properties were evaluated by the loss tangent maximum (tan δ_{max}) and the temperature range (ΔT) for efficient damping (tan δ > 0.3).

Thermogravimetric Analysis (TGA)

A TA instrument thermogravimeter, Q5000 IR (TA Instruments, New Castle, DE, USA) was used to measure the weight loss of polymers in air (20ml/min). Samples were heated from 30⁰C to 800⁰C at a heating rate of 20⁰C /mm.

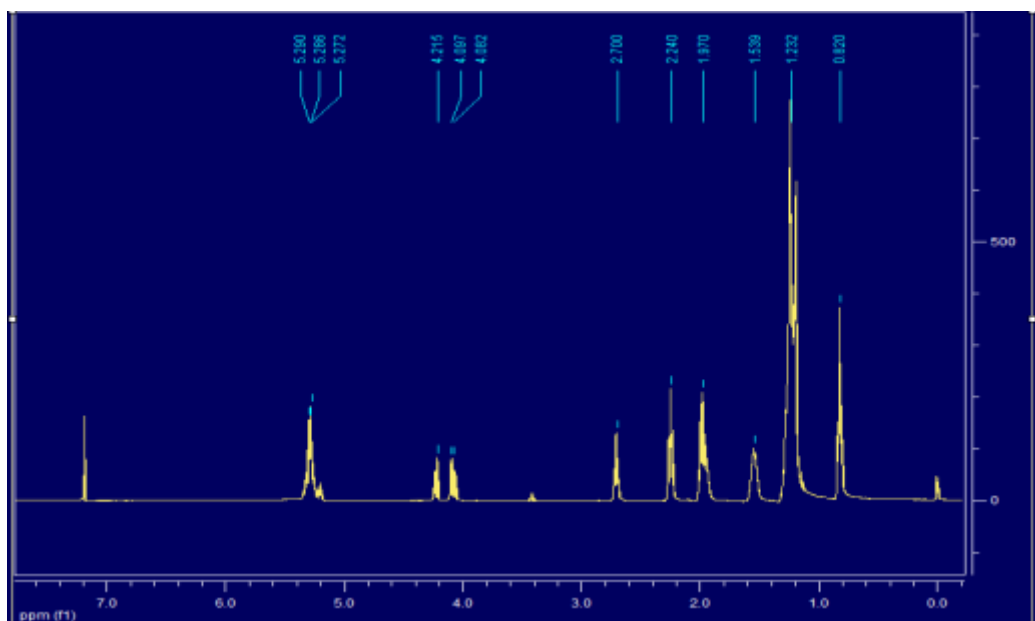
Mechanical Test

The compressive mechanical tests were conducted according to DIN EN ISO 527 specification using a Zwick universal testing machine (Zwick GmbH and Co, KG, Ulm, Germany) at a cross head speed of 2mm/min. The tensile modulus (Et), tensile stress (σ_m), yield or failure stress (σ_b) and elongation at break (ε_b) were obtained from the tensile tests.

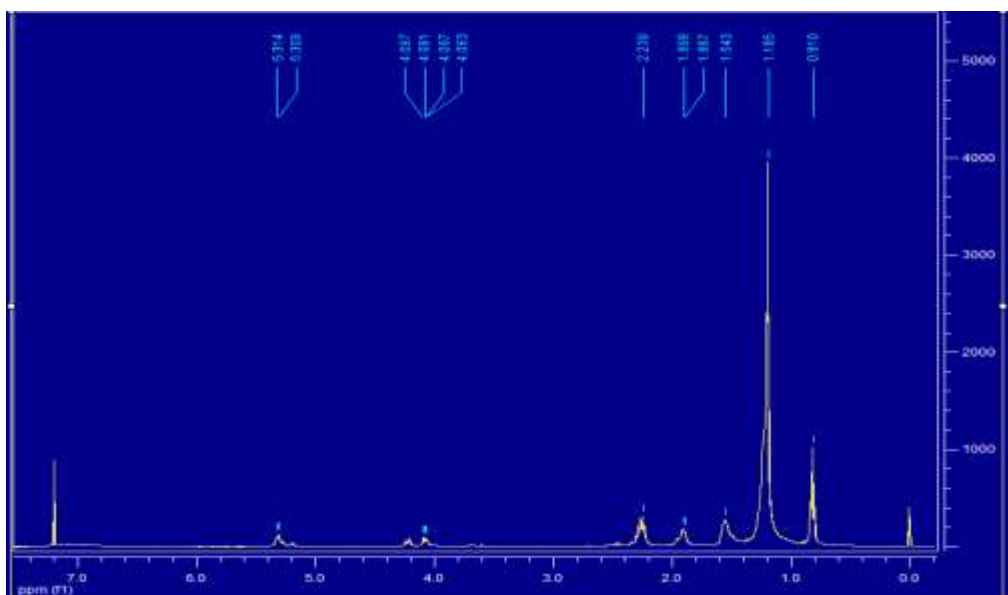
Results and Discussion

Molecular structure of ADBO and microstructures of resulting thermosetting polymers from ADBO

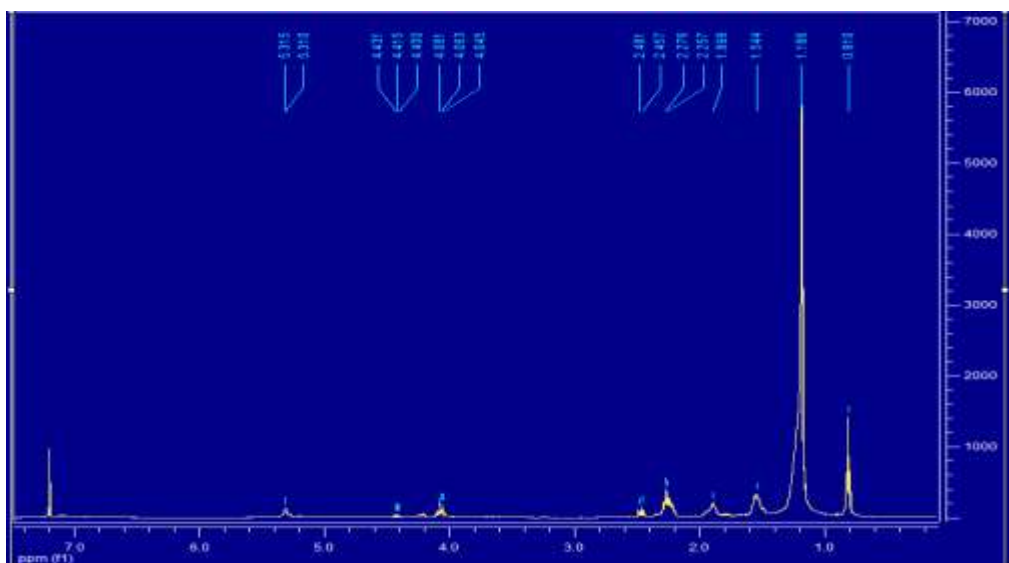
A variety of polymeric materials ranging from soft rubbers to tough and rigid plastics were obtained from the cationic polymerization of ADBO with styrene and divinylbenzene and the yields were found to be quantitative. The different polymers were soxhlet extracted using methylene chloride as refluxing solvents and the extractables were studied using ^1H NMR in order to understand the structure of the materials. Generally, about 72 – 98 wt% of insoluble substances were retained after extraction of bulk polymers Figure 1a presents the ^1H NMR spectra of ADBO. The peaks at 4.1- 4.4ppm and 5.2 - 5.5ppm originated from the methylene protons and vinylic protons of the triglyceride. The protons of CH_2 groups sandwiched between two carbon – carbon double bonds appeared at 2.7-2.8ppm. Figure 1b and 1c present the ^1H NMR of extractables from ADBO55 – ST25 – DVB12 – MC8 and ADBO45 – ST22 – DVB25 – MC8. It is observed that the extracted soluble substances are composed of catalytic initiator fragment ($\approx 4.2\text{ppm}$), triglyceride protons (4.2 – 4.5ppm) of the oil and weak vinylic protons peaks at 5.2 – 5.5ppm indicating that the extracted oil are much less unsaturated than the ADBO originally used. The absence of peaks in the region 6.4 – 7.5ppm in the spectra 1b and 1c indicate that all of the aromatic monomers were incorporated in the crosslinked polymer networks.



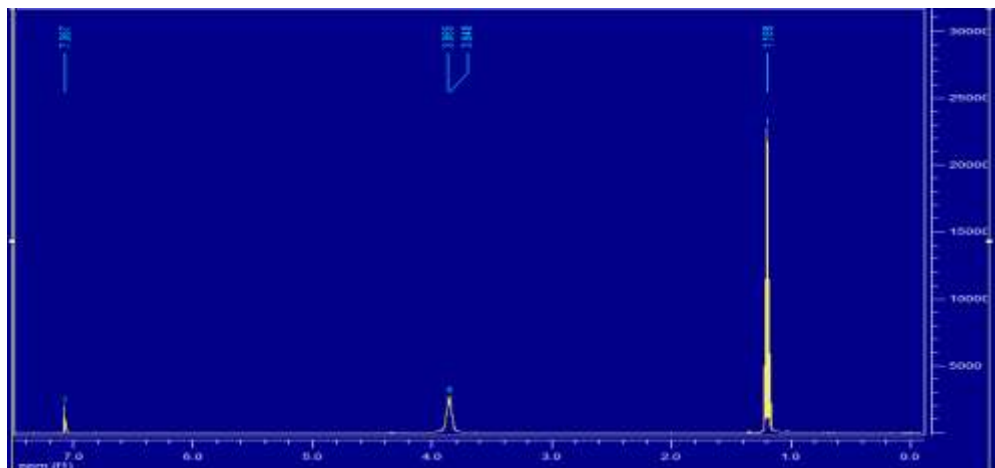
a



b



c



D

Figure 1 Proton NMR spectra of (a) *Adenopus breviflorus* oil, (b) soluble materials from ADBO55 – ST25 – DVB12 – MC8 (c) soluble materials from ADBO45 – ST22 – DVB25 – MC8 and (d) pure BF3E

Based on the fatty acid composition of the ADBO earlier reported by us, Akintayo and Bayer [9] and using the formula;

$$\text{Mole of double bonds/mole of Triglyceride} = \frac{\sum \text{mole of double bond}/100\text{g}}{\sum \text{mole of fatty acid}/100\text{g}} \times 3$$

Table 1 Fatty acid composition, moles of double bond and fatty acid per 100g of oil for *Adenopus breviflorus* oil.

	% Fatty acid ^a	Molar mass	Number of double bonds	Moles of double bond/100g solution	Moles of fatty acid/100g oil
Oleic	13.8	282.4614	1	0.0489	0.0489
linoleic	61.3	280.4500	2	0.4372	0.2186
linolenic	-	278.4500	3	0	0
Steoric	14.1	284.4800	0	0	0.501
palmitic	10.8	256.4200	0	0	0.0421
Σ				0.4861	0.3597

a- Akintayo E.T and Bayer.E(2002). Biores. Tech.85, 95-97

The ADBO oil is calculated to have 4.1 carbon-carbon double bonds per triglyceride as presented in table 1.0. This value is same as that in corn oil [10] and close to 4.5 C=C bonds in soybean oil [5] which have successfully been used to prepare thermosetting polymers.

Table 2.0 lists the composition of the bulk polymers prepared by varying the ADBO concentration. It is generally observed that when ADBO concentration increased, the yield of the insoluble substance (i.e. the cross linked polymer) decreased from 82.4% to 72.3%.

Table 2 Appearance and Soxhlets extraction results

Entry	Polymer	Product appearance	Extraction (wt. %)	
			Insoluble	Soluble
1	ADBO30-ST37-DVB25-MC8	Hard, ductile, plastic	82.4	17.6
2	ADBO45-ST28-DVB19-MC8	Rigid, ductile, plastic	78.9	21.1
3	ADBO55-ST22-DVB15-MC8	Soft, ductile, plastic	72.3	27.7
4	ADBO45-ST27-DVB20-MC8	Hard, ductile, plastic	82.4	17.6
5	ADBO45-ST25-DVB22-MC8	Hard, ductile, plastic	88.6	1.4
6	ADBO45-ST22-DVB25-MC8	Hard, ductile, plastic	91.4	8.6

Table 2, entries 2, 4, 5 and 6 also show that increasing DVB concentration, generally increased the yield of cross-linked polymers, thereby, indicating DVB as an effective cross-linking agent. The ADBO – ST – DVB copolymers ranged from hard to soft plastics as the oil content increased (Table 2.0, entries 1,2 & 3) while the ADBO – ST – DVB copolymers were all hard but ductile plastics (Table 2, entries 4,5 & 6).

Dynamic Mechanical Properties of ADBO co-polymers.

Figure 2 shows the temperature dependence of the storage modulus of the ADBO copolymers prepared by varying the ADBO concentration while keeping the ST: DVB ratio constant.

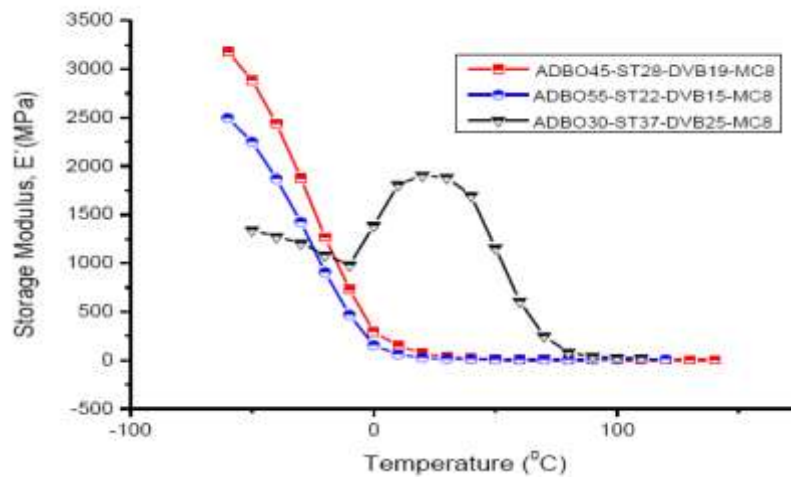


Figure 2 Temperature dependence of the storage modulus of ADBO polymers prepared by varying the ADBO concentration while ratio of ST: DVB remained constant at 3:2

Figure 3 however presents the temperature dependence of the storage modulus of the ADBO copolymers while varying the DVB concentration but keeping the ADBO concentration constant.

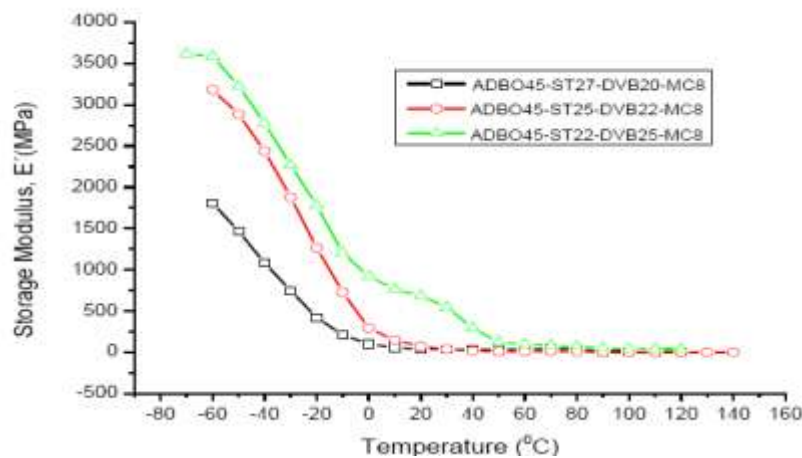


Figure 3 Temperature dependence of the storage modulus of ADBO polymers prepared by varying the DVB concentration while the content of ST plus DVB remained constant at 47%

Generally, the storage modulus exhibited a decrease over a wide temperature range, followed by a modulus plateau at high temperatures where the polymers behaved like rubber. Li and Larock [11] had explained that modulus drop corresponds to the onset of segmental mobility in the polymer chain and the appearance of relatively stable modulus at high temperature (as observed in this work) indicates the existence of stable cross-links in the bulk polymer. Figure 2 shows that

ADBO30 – ST37 – DVB25 – MC8 had high storage moduli over a wide range of temperature. This may be attributed to the predominance of rigid ST+DVB segments in the cross-linked polymer backbone. As the ADBO concentration increased, the resulting polymers possessed decreasing amount of ST + DVB segments.

Figure 3 shows the temperature dependence of storage modulus E' for the ADBO polymers prepared by varying the concentration of DVB when the total concentration of the co-monomers of ST and DVB was kept at 47wt%. The ADBO45 – ST27 – DVB20 – MC8 polymer exhibited low moduli compared to other copolymers containing DVB22 and DVB25 respectively. An increase in DVB concentration therefore led to an increase in the E' . Such behaviour was expected as increasing the DVB concentration increases the degree of cross-linking.

Based on the kinetic theory of rubber elasticity, the experimental crosslink densities of all copolymers (V_e) can be determined from the rubbery moduli using the following equation:

$E' = 3 V_e RT$ [14] where E' is the storage modulus of the cross linked copolymers in the rubbery plateau region above the glass transition temperature (T_g) [12, 13], R is the universal gas constant and T is the absolute temperature. The crosslink densities (V_e 's) calculated for the different co-polymers using the above formula is presented in table 3.0. The result show a slight decrease in V_e as the crosslinking agent (DVB) concentration decreased (entries 1 – 3) but a significant increase in V_e as the DVB concentration increased (entries 4 –6).

Damping Effect and Storage memory Properties

Figures 4 and 5 present the temperature dependence of the loss factor, $\tan \delta$ and loss modulus respectively for ADBO copolymers prepared by varying the ADBO concentration while the ratio of ST:DVB remained constant at 3:2.

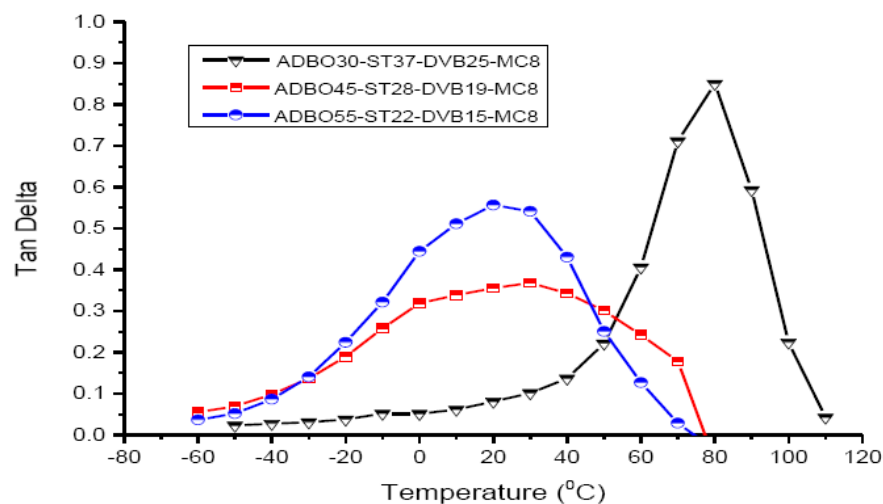


Figure 4 Temperature dependence of the loss factor, $\tan \delta$ of ADBO polymers prepared by varying the ADBO concentration while ratio of ST: DVB remained constant at 3:2

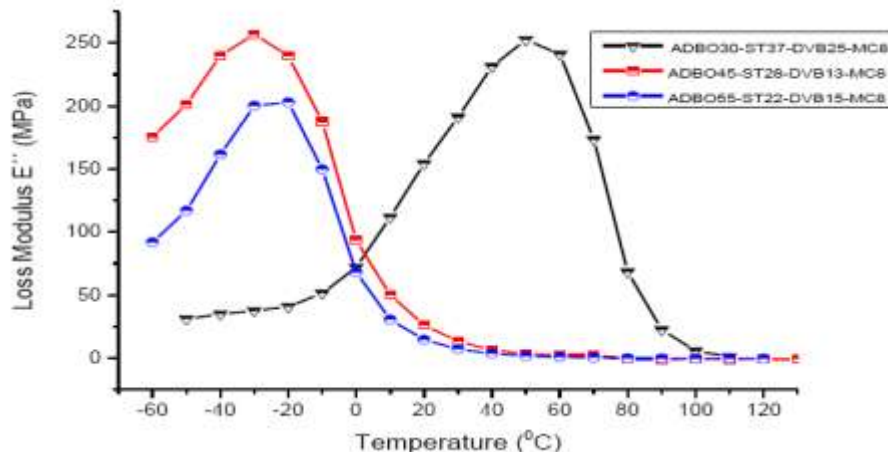


Figure 5 Temperature dependence of the loss modulus of ADBO polymers prepared by varying the ADBO concentration while ratio of ST: DVB remained constant at 3:2

Li et al. [10] had reported that a good damping material should possess a high $\tan \delta$ ($\tan \delta > 0.3$) over a temperature range ΔT of at least $60 - 80^{\circ}\text{C}$. Ester groups attached to the polymer structures of vegetable oil based have been reported to contribute greatly to their damping properties [15, 13, 16]. The $\tan \delta$ which indicates the damping ability of the co-polymer over certain temperature range ΔT and the glass transition temperature T_g obtainable from DMA analysis ($\tan \delta$ plot) are presented in table 3.0 The $\tan \delta$ is the ratio of mechanical dissipated energy to the storage energy. Therefore a high $\tan \delta$ value is essential for good damping properties. Entries 1 – 3 in table 3.0 show that as the ADBO concentration increased, $\tan \delta_{\text{max}}$ of the resulting ADBO polymers varied from 0.37 to 0.85 but only over a temperature range of 25°C to 67°C . Compared to the results obtained for copolymers prepared from other vegetable oils the ADBO co-polymer could be said to have good damping properties. Entries 4–7 in table 3.0 summarize the effect of DVB concentration on the damping properties of the resulting ADBO co-polymer.

ADBO 45-ST 28-DVB 19-MC8 polymer exhibited a $\tan \delta_{\text{max}}$ value of 0.37 and a ΔT of 25°C at $\tan \delta > 0.3$. However as DVB concentration increased (entries 5 – 7) the $\tan \delta_{\text{max}}$ decreased to the vicinity of 0.30 over a much smaller temperature range and the copolymers were no longer good damping materials compared to those of entries 1 – 3.

The ADBO co-polymer could also be considered to show shape memory behavior. According to Li and Larock [17], a shape memory polymer basically possesses a certain degree of crosslink and a T_g higher than room temperature. The results of T_g 's as presented in table 3.0 varied from $\approx 21^{\circ}\text{C}$ to 80°C for entries 1 – 3 and from $\approx 13^{\circ}\text{C}$ to 59°C for entries 4 – 7 and the thermosets with T_g 's higher than room temperature are expected to exhibit shape memory behavior. As the ADBO concentration increased from 30% to 55% the T_g 's of the resulting polymers decreased from 80.47°C to 20.86°C indicating that polymers ranging from rubbery materials to hard plastics were produced. However, as the DVB concentration increased from 20 to 25% and ADBO concentration kept constant, (entries 5, 6 and 7) the T_g 's of the resulting polymer increased from

12.9⁰C to 58.54⁰C indicating that rubbery materials to rigid plastics have been produced by varying the DVB concentration. Also observable from Figures 6 and 7 is the fact that as DVB concentration increased, the molecular motions on the resulting copolymer became more restricted and the amount of energy that could be dissipated throughout the polymer decreased. Therefore the tan δ peak positions of the polymer shifted to higher temperature.

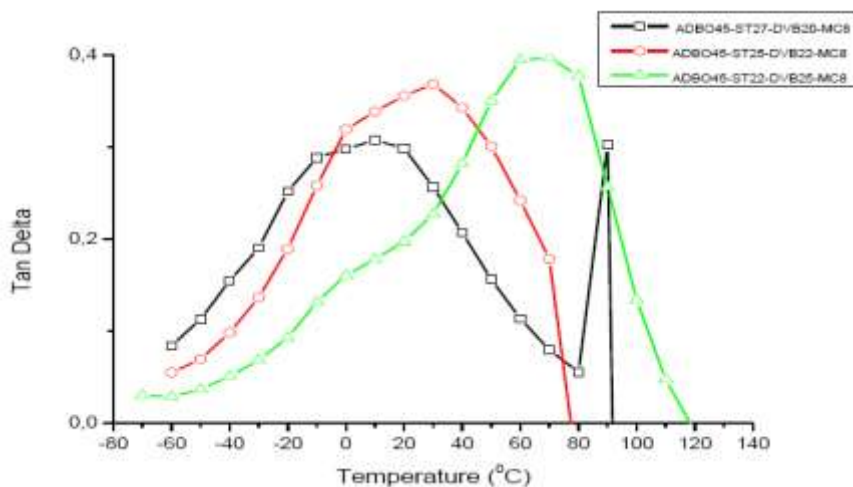


Figure 6 Temperature dependence of the loss factor, tan δ of ADBO polymers prepared by varying the DVB concentration while the content of ST plus DVB remained constant at 47%

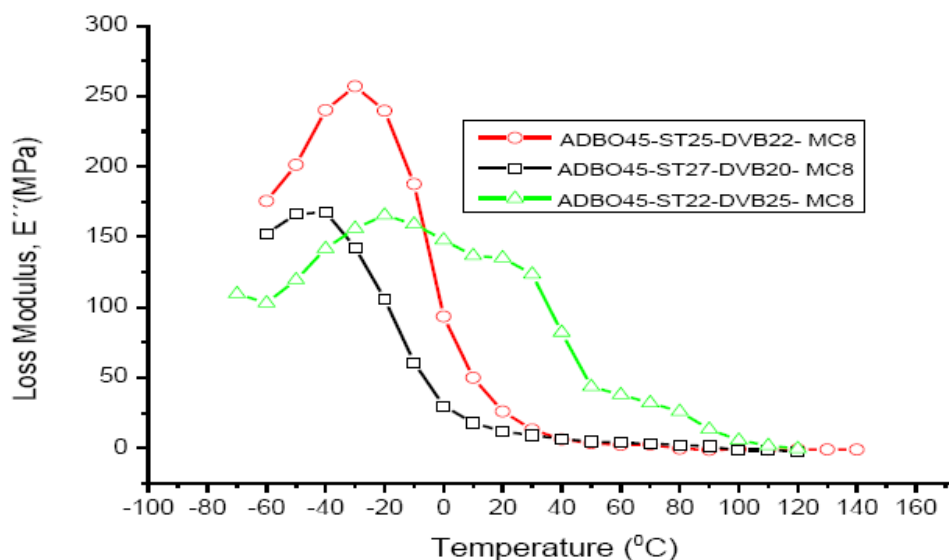


Figure 7 Temperature dependence of the loss modulus of ADBO polymers prepared by varying the DVB concentration while the content of ST plus DVB remained constant at 47%

Table 3 DMA Characteristics of ADBO co-polymers

Entry	Polymer	T _g (°C)	V _e (mol/dm ³)	Tan δ _{max}	ΔT
1	ADB030-ST37-DVB25-MC8	80.47	2.98X 10 ³	0.85	44
2	ADBO45-ST28-DVB19-MC8	31.42	2.90 X 10 ³	0.37	25
3	ADBO55-ST22-DVB15-MC8	20.86	2.39 X 10 ³	0.57	67
4	ADBO45-ST28-DVB19-MC8	31.42	2.9 X 10 ³	0.37	25
5	ADBO45-ST27-DVB20-MC8	12.90	6.60 X 10 ³	0.31	25
6	ADBO45-ST25-DVB22-MC8	45.90	6.28 X 10 ³	0.30	8
7	ADBO45-ST22-DVB25-MC8	58.54	20.92 X 10 ³	0.32	13

Table 4 Mechanical Properties of the ADBO co-polymers

Entry	Polymer	Tensile tests			
		E _t (MPa)	σ _m (MPa)	σ _b (MPa)	ε _b (MPa)
1	ADBO30-ST37-DVB19-MC8	404.23	7.87	7.87	2.77
2	ADBO45-ST28-DVB19-MC8	10.25	1.77	0.86	27.38
3	ADBO55-ST22-DVB25-MC8	1.47	0.27	0.18	34.02
4	ADBO45-ST28-DVB19-MC8	10.25	1.77	0.86	27.38
5	ADBO45-ST27-DVB20-MC8	22.84	1.53	1.53	31.02
6	ADBO45-ST25-DVB22-MC8	24.77	2.52	2.52	20.26
7	ADBO45-ST22-DVB25-MC8	97.85	4.21	4.21	19.65

E_t =Tensile Modulus; σ_m =Tensile stress; σ_b = Yield or failure stress; ε_b =Elongation at break

Mechanical properties

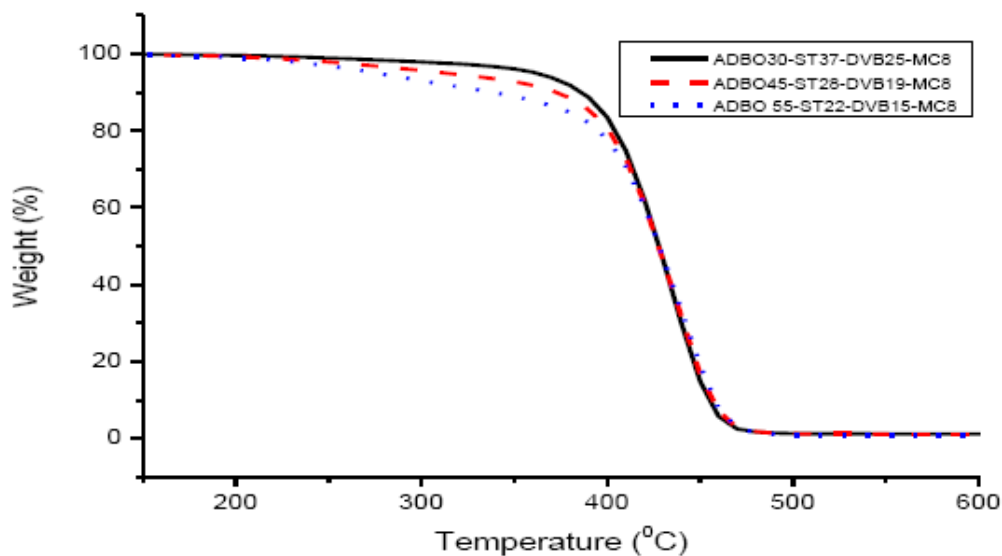
The tensile mechanical properties of the ADBO polymers are presented in Table 4. The ADBO30-ST37-DVB25-MC8 which contained considerable amounts of rigid aromatic co-monomer showed the typical tensile behavior of a plastic with tensile modulus (E_t) = 404.23MPa and yield stress ($\bar{\sigma}_b$) = 7.87MPa (entry1). As the amount of ADBO increased, the resulting polymers (entry 2) showed a big decrease in the E_t and $\bar{\sigma}_b$. However, their elongation at break ϵ_b increased showing that as ADBO concentration increased, the ductility of the resulting polymer increased. When the ADBO concentration exceeded that of the ST + DVB co-monomers, the resulting polymer, ADBO55-ST22-DVB15-MC8 (entry 3) exhibited a tensile behavior similar to that of a soft rubbery material. Entries 4-7 in table 4.0 also show that the tensile mechanical properties of ADBO co-polymers were also greatly affected by the DVB concentration. As the DVB concentration increased the E_t and $\bar{\sigma}_b$ of the resulting polymers increased while their elongation at break ϵ_b appeared to decrease overall.

Thermogravimetric Analysis

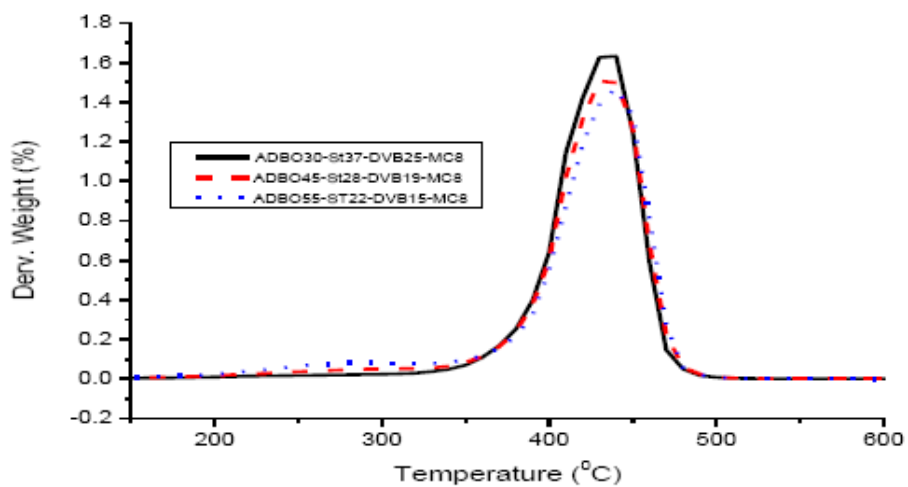
The TGA thermograms of the ADBO co-polymers with increasing ADBO concentration while keeping the ST : DVB ratio 2 :3 and that of ADBO polymers with increasing DVB concentration while maintaining the ST + DVB content at 47% are presented in Figures 8a and 9a respectively. Their TGA derivative curves are also presented in Figures 8b and 9b respectively, and the TGA data are summarized in table 5. The thermograms indicate that the polymer exhibit a three stage thermal degradation step. The first stage degradation (200⁰C to 400⁰C) may be attributed to evaporation and decomposition of unreacted oil and other soluble components in the bulk material. The second stage occurred between 400 and 500⁰C and it was a very rapid stage corresponding to degradation and char formation of the crosslinked polymer. The last stage which occurred at around 500 ⁰C and above may be attributed to gradual oxidation of the char residue. Results in table 5 indicate that as ADBO concentration increased (entries 1 -3) the onset of first degradation step decreased, presumably because of decreasing crosslink density as ADBO concentration increased while as the DVB concentration increased (entries 4, 5 and 6) the onset temperature of first degradation step increased. ADBO co-polymers in entries 1-3 lose between 5.9 – 11.9% of their weight at temperatures varying from 215 to 348⁰C and lose between 87 – 93% weight between 405 and 459⁰C. For polymers with increasing DVB content (entries 4, 5 and 6) they lose between 3.5 – 6.5 wt% between 200 – 357⁰C and 91 – 93% between 401 – 457⁰C. The Tmax which is determined as the maximum of the corresponding derivative weight % loss curve only varied slightly among the samples and indicate that the ADBO copolymers exhibit similar thermal behaviours.

Table 5 TGA data of ADBO co-polymers

Entry	Polymer	Step Transition 1			Step Transition 2			Step transition 3			T _{max}
		Onset(°c)	Endset(°c)	Wt(%)	Onset(°c)	Endset(°c)	Wt(%)	Onset(°c)	Endset(°c)	Wt(%)	
1	ADOBO30-ST37-DVB25-MC8	228	335	5.91	407	490	2.92	502	525	0.38	434.84
2	ADOBO45-ST28-DVB19-MC8	220	329	7.92	405	456	0.52	496	516	0.18	442.61
3	ADOBO55-ST22-DVB15-MC8	215	348	11.8	407	456	2.16	499	525	0.49	441.38
4	ADOBO45-ST27-DVB20-MC8	200	320	6.3	401	455	1.45	504	516	0.42	427.40
5	ADOBO45-ST25-DVB22-MC8	229	321	5.6	401	455	1.13	497	519	0.83	435.66
6	ADOBO45-ST22-DVB25-MC8	235	357	3.5	411	457	2.87	499	526	0.62	441.71

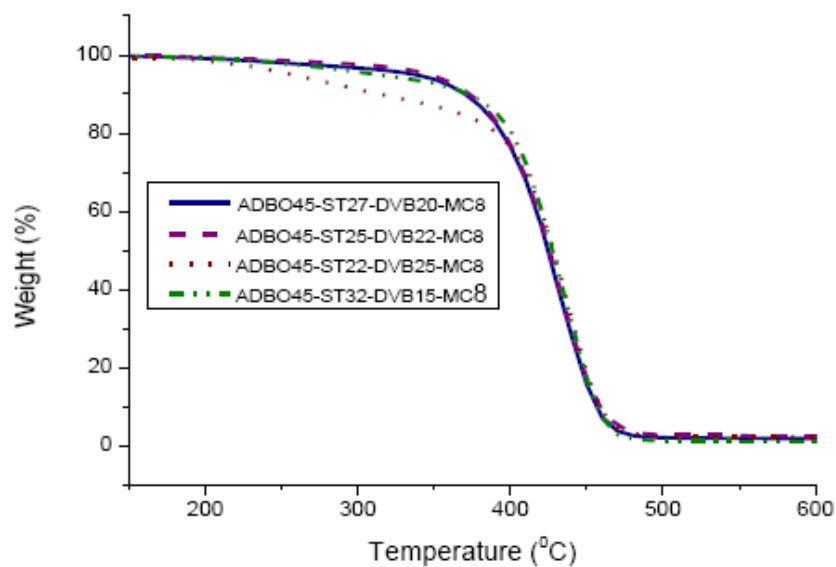


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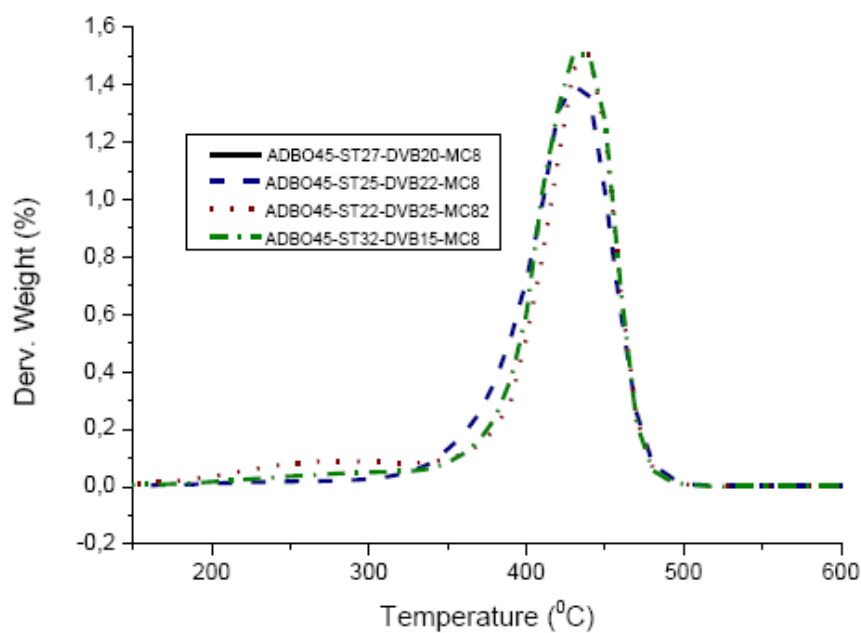


b

Figure 8 (a) TGA curves of ADBO polymer with increasing ADBO concentration; (b) TGA derivative curves of ADBO polymers with increasing ADBO concentration.



a



b

Figure 9 (a) TGA curves of ADBO polymers with increasing DVB concentration; (b) TGA derivative curves of ADBO polymers with increasing DVB concentration.

Conclusion

Adenopus breviflorus oil was effectively copolymerized with styrene and divinylbenzene to give viable thermosets by cationic polymerization initiated by boron trifluoride etherate. The thermosets ranged from soft rubbers to hard plastics depending on the stoichiometry employed. DMA shows the resulting materials as possessing crosslink densities ranging between 2.39×10^3 to $2.09 \times 10^4 \text{ mol/m}^3$ and a single glass transition temperatures ranging between $12 - 80^\circ\text{C}$. The thermosets were thermally stable below 200°C and exhibited three stages of degradation, generally between $200 - 400^\circ\text{C}$; $400 - 500^\circ\text{C}$ and $> 500^\circ\text{C}$. The tensile stress – strain behavior of the newly prepared thermosets were also found to depend on the stoichiometry of the component parts.

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