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Synthesis and Characterization of Anethole-Lauryl Methacrylate Copolymer via Cationic Polymerization

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ABSTRACT. The synthesis of anethole-lauryl methacrylate (LMA) copolymer had been carried out by cationic polymerization employing $BF_3O(C_2H_5)_2$ as the initiator without the use of solvent at room temperature (28-30 °C) over atmospheric N_2 conditions. Polymerization are made with anethole variations of 2%, 4%, and 16% against LMA weight. Structural determination of co-poly(anethole-LMA) was done using FTIR and ^{1}H-NMR spectroscopy. The relative molecular weight (M_v) of co-poly (anethole-LMA) was determined by an Ostwald Viscometer at room temperature. Morphological characterization and surface area analysis of co-poly(anethole-LMA) was carried out using SEM and SAA (Surface Area Analyzer), respectively. The successful synthesis of co-poly(anethole-LMA) was proven through the disappearance of vinyl group absorption at 1696, 1638, 965, and 938 cm^{-1} of the FTIR spectra, as well almost loss of vinyl group proton signals at 6.4-5.5 ppm in the ^{1}H-NMR spectra. Increasing the weight of the LMA affected the characteristics of co-poly(anethole-LMA). The relative molecular weight of co-poly(anethole-LMA) was found to rise by increasing the weight of LMA. The M_v of co-poly(anethole-LMA) 2%, 4%, and 6% were 32378.62, 50611.05, and 65133.79 g/mol, respectively. The morphology of co-poly(anethole-LMA) displayed that the surface distance between LMA and anethole particles was getting tighter and the highest surface area in co-poly(anethole-LMA) 6% was 233.80 m^2/g.

Keywords : anethole, boron trifluoride etherate, cationic polymerization, lauryl methacrylate

INTRODUCTION

Anethole, 1-Methoxy-4-[(1E)-prop-1-en-1-yl]benzene, is the main constituent of fennel oil (*Foeniculum vulgare Mill.*) and anise oil (*Pimpinella anisum L.*) (Prakosa et al., 2013). Anethole contained in fennel oil is 50-80%, while anethole contained in anise oil is 80-95% (Kardinan & Dhalimi, 2016; Mohammed, 2009). Anethole has a reactive group, namely the propenyl group which is very potential if modified into other compounds that are more useful (Zhou et al., 2018). The reactive group contained in anethole is modified to increase its used value, for example as a polymeric material.

LMA (lauryl methacrylate), a hydrophobic monomer that is soluble in non-polar solvents (Raghunadh et al., 2004), has an alkyl group in its long chain, which can provide a polymer with great flexibility having low glass transition temperature, T_g (Ahmad et al., 2011; Shabnam & Ahmad, 2016). LMA can produce polymers with better thermal and mechanical properties when these compounds are cross-linked (Shabnam & Ahmad, 2015). The resulting polymers were reported to have good potential applications for example Wang et al., (2022) innovatively synthesized LMA copolymer with divinylbenzene (DVB) as the crosslinker by suspension polymerization for oil absorption. Other studies have also shown that the polymerization of LMA graft to bamboo fibers has been successfully applied to oil spills with an absorption capacity of 20.0 g of oil/g sorbent (Tung et al., 2022).

Polymers have an important role in human life in this modern world because polymers have many advantages compared to other traditional materials. The properties of polymeric materials include higher energy efficiency, lighter weight, and higher performance and durability (Erdmenger et al., 2010). Currently, most of the polymers produced are of petroleum origin which is non-renewable, environmentally unfriendly, and non-biodegradable. This results in polymer waste that has accumulated for decades which can damage the environment (Bhadra et al., 2017). The solution that can be done to overcome this problem is that polymer base materials are needed from biological resources that are renewable and environmentally friendly. Anethole can be an alternative polymer material that is expected to have the same or even better performance than petroleum-based materials so that it can be used to modify polymer materials to produce environmentally friendly polymers (Tao et al., 2017).

The polymerization process is divided into two types, namely step (condensation) and chain (addition) polymerization. Monomers that can undergo addition polymerization must have double bonds (C=C) in their functional groups (Tomar et al., 2016). The structure of anethole is similar to styrene and eugenol which have one aromatic ring and one vinyl group which could be polymerized (Handayani et al., 2019). Anethole can be used as a starting material for cationic polymerization, including the type of addition polymerization. Chain carriers in a reaction through cationic polymerization are carbonium ions obtained by the addition of an electrophile initiator (Hamzah, 2017; Lena et al., 2020). Baghdadi et al (2016) argued the initiators used in cationic polymerization are *Lewis* acid and *Bronsted* acid these have been applied in the cationic polymerization process to styrene. *Lewis* acids can be used, such as $BF_3O(C_2H_5)_2$, $AlCl_3$, $SnCl_4$, PCl_5, $SbCl_5$, $TiCl_4$, and $RAlCl_2$, while *Bronsted* acids that can used as initiators as H_2SO_4 and HNO_3. Initiators that can be used in cationic polymerization include (Pitsikalis, 2016).

This study was conducted to produce an anethole-based polymer to increase the used-value of anethole. Anethole is structurally similar to styrene and eugenol, which could be polymerized with LMA through cationic polymerization with the $BF_3O(C_2H_5)_2$ initiator. The polymers produced by this method are expected to improve the quality of the polymer and produce polymers that are environmentally friendly as well as can be used more widely.

EXPERIMENTAL SECTION

Materials

Anethole 91.94% was obtained from GC Test Results of Schimel Rect-DAB fennel oil. LMA (Lauryl Methacrylate) was afforded from Sigma Aldrich and prior to polymerization, it was shaken by employing a 5% of NaOH, distilled water, and subsequently dried over anhydrous Na_2SO_4. Boron trifluoride etherate ($BF_3O(C_2H_5)_2$), diethyl ether, chloroform, methanol, universal pH paper, and Na_2SO_4 anhydrous were obtained from Merck. Nitrogen was purchased from the Samator industry.

Synthesis of Co-poly(Anethole-LMA)

Anethole as much as 2 g and LMA through a weight variation of 2%, 4%, and 6% (w/w) of anethole weight was put into a 100 mL three-neck flask. The polymerization process was performed by flowing nitrogen gas at r.t. The two monomers were stirred with a magnetic stirrer for 15 minutes to form a mixture. The copolymerization was proceeded for 24 h. with the addition of $BF_3O(C_2H_5)_2$ initiator which was carried out twice in the first 2 h, each as much as 0.05 mL. The polymerization was stopped by adding 0.1 mL of methanol. The resulting copolymer was then dissolved in distilled water to be followed by the extraction of diethyl ether until its pH became 7. The organic layer was collected, dried over anhydrous Na_2SO_4, and evaporated. Product characterization was done using FTIR and ^{1}H-NMR spectrophotometers.

Calculated M_v of Co-poly(Anethole-LMA)

The M_v determination of co-poly(anethole-LMA) was carried out by measuring the flow time using an Ostwald viscometer in various concentrations i.e. 0.00125, 0.0025, 0.005, and 0.01 g/mL as well as pure chloroform. The flow time determination was performed at r.t. The obtained flow time data of co-poly(anethole-LMA) solution and pure chloroform solvent were then used to calculate the relative viscosity (ηr) and specific viscosity (ηsp). The $\eta sp/C$ versus C was then plotted to obtain an intrinsic viscosity $[\eta]_i$. The M_v was computed by the Mark-Houwink equation, by means of $K = 0.011$ and $a = 0.725$.

FTIR (Fourier Transform Infra Red) Analysis

FTIR analysis was performed on Shimadzu FTIR Prestige-21 spectrophotometer in the range of 4000-500 cm^{-1}. The sample was prepared as KBr pellets and the spectra were recorded at r.t. The resulting film was directly attached to the sample holder.

^{1}H-NMR (Nuclear Magnetic Resonance) Analysis

^{1}H-NMR analysis was done on Agilent NMR 500 MHz. The spectra were taken using $CDCl_3$ at r.t. and the chemical shift was described in δ (ppm) using TMS as an internal standard. The coupling constant (*J*) was reported in Hz.

Morphological Analysis of Co-poly(Anethole-LMA)

The resulting SEM data used is the surface morphology of (Anethole-LMA) with a magnification of 2500 times. The morphology of the co-poly(Anethol-LMA) is visually analyzed. The effect of the number of additions by weight percentage of LMA from anethole weight compared to the morphology of co-poly(Anethole-LMA) 2%, 4%, and 6% (w/w).

Surface Area Analysis of Co-poly(Anethole-LMA)

The surface area of copolymer (Anethole-LMA) 2%, 4%, and 6% (w/w) can be analyzed using the Brunauer, Emmett, Teller (BET) method with nitrogen gas (N_2) as adsorbate, with a degassing temperature of 40 °C, sample testing time of 6 o'clock. This analysis is based on physical adsorption isotherm data. Equilibrium isotherms can describe where the adsorbed volume is plotted against P/P_o (P: pressure, P_o: saturation pressure at measurement temperature) shown in the BET method.

RESULTS AND DISCUSSION

Synthesis of Co-poly(Anethole-LMA)

The polymerization of anethole and LMA monomers was performed by cationic polymerization using the $BF_3O(C_2H_5)_2$ initiator without solvent at room temperature and under the nitrogen atmosphere. The initiator $BF_3O(C_2H_5)_2$ turns as a catalyst which speeds up the reaction by producing active groups to extend the polymer chain (Manurung et al., 2013). Synthesis was accomplished under a nitrogen atmosphere at r.t. (28-30 °C). that is because the cationic polymerization process is sensitive to air and the presence of nitrogen gas can expel water vapor and other proton-giving molecules that can react with the initiator. The presence of water will cause a hydrolysis reaction to the initiator $BF_3O(C_2H_5)_2$ so that the initiator becomes inactive and results in a decrease in the reaction rate of polymer chain formation (Destephen et al., 2020; Huang et al., 2007).

The co-poly(Anethole-LMA) reaction was carried out for 24 hours with the addition of an initiator gradually every 1 hour in the first 2 hours. Cationic polymerization without solvent media has several advantages process is easy, more efficient, and more economical. Anethole monomers and LMA have a similar phase, which is liquid make they can directly react through cationic polymerization without solvent media. Cationic polymerization is an addition reaction involving a double bond (C=C) on the vinyl group of each monomer to a single bond (C-C) due to the addition of an initiator. The successful polymerization can be analyzed by FTIR and ^{1}H-NMR. The synthesis of co-poly (Anethole-LMA) on the weight of the LMA variation afforded the product in the form of solids. The weight and %yield of all variations are offered in **Table 1.** The physical form of co-poly (anethole-LMA) produced from these three variations has the same shape and color, that is powder-shaped and yellowish-white. The data presented in table 1 shows that the more weight percentage of LMA added, the less copolymer yield obtained. That is because LMA has a long carbon chain that has been propagating, causing a steric factor that will inhibit the copolymer propagation rate. The decrease in yield from 4% co-poly(anethole-LMA) to 6% co-poly(anethole-LMA) was relatively good than the decrease in yield from 2% co-poly(anethole-LMA) to 4% co-poly(Anethole-LMA). That is because at the stage of dissolving co-poly(anethole-LMA) 6% with diethyl ether there is a white precipitate. Such white deposits are possible as residual reactions between anethole monomers or LMA that do not react as a whole due to the presence of steric factors.

The cationic polymerization mechanism consists of 3 stages; (1) initiation (2) propagation and (3) termination (Hamzah, 2017). The initiation stage occurs after the addition of the $BF_3O(C_2H_5)_2$ initiator to the anethole and LMA mixture. The initiator formation of $BF_3O(C_2H_5)_2$ is through a covalent bond between the O atom and the B atom, which comes from BF_3. The lone pair electron used to bond comes from the O atom so that the $-C_2H_5$ group will tend to be positive partial (cation). The resulting $(C_2H_5)^+$ cation will start the initiation process by adding the vinyl double bond to the anethole and LMA to form a carbocation, as shown in **Figures 1** (a) and (b). Based on the research, at this initiation stage, the anethole might have been first initiated because the anethole structure had a smaller steric than the LMA and the anethole weight ratio was greater than the LMA.

Table 1. The Results of co-poly(anethole-LMA) synthesis

Copolymer	Initial mass (g)		Product (g)	Yield (%)
	Anethole	LMA		
Co-poly(Anethole-LMA) 2%	1.838	0.037	1.776	94.72
Co-poly(Anethole-LMA) 4%	1.838	0.074	1.755	91.79
Co-poly(Anethole-LMA) 6%	1.838	0.110	1.555	79.83

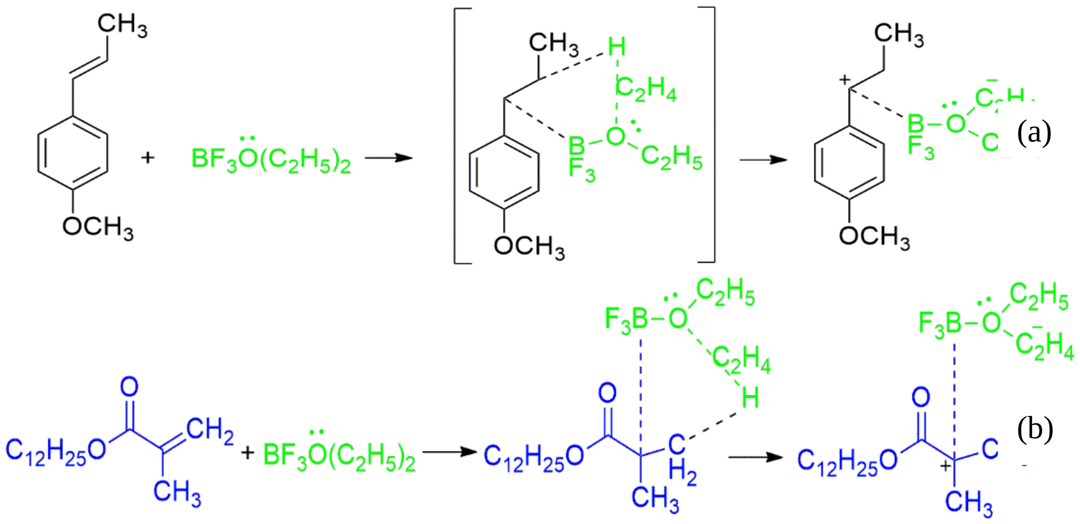

Figure 1. Probable initiation reaction on (**a**) Anethole and (**b**) LMA.

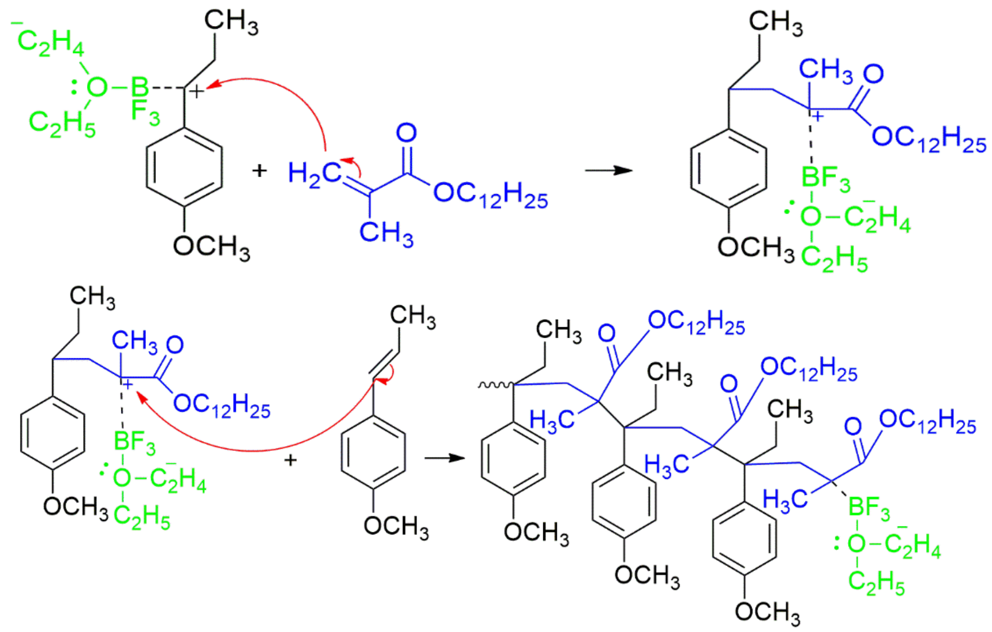

Figure 2. Suggested propagation reaction on co-poly(anethole-LMA) synthesis

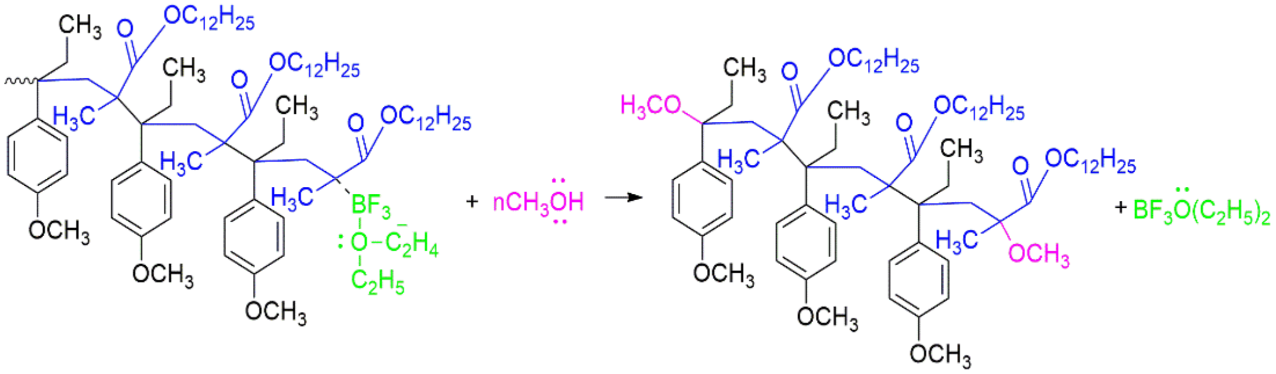

Figure 3. Possible termination reaction on co-poly(Anethole-LMA) synthesis

The stability of the carbocation formed at the initiation stage might affect the propagation rate (Sudarlin & Haryadi, 2017). The C=C bond in the LMA would attack the carbocation in the anethole which could initiate and form a new carbocation. This process is going repeatedly until the chain extends (propagation stage) as depicted in **Figure 2**. The termination stage aims to stop the polymerization rate by adding methanol (CH_3OH). The oxygen atom in methanol is a nucleophile which could cause the carbocation at the end of the long copolymer chain to become inactive so that the rate of propagation could stop. The proposed propagation reaction is displayed in **Figure 3.**

FTIR Analysis

The successful co-poly(anethole-LMA) synthesis was analyzed through FTIR spectra by comparing the absorption characteristics of functional groups of the anethole, LMA, and co-poly(anethole-LMA), as depicted in **Figure 4**. FTIR spectral analysis by comparing the absorption characteristics of the functional groups of each monomer and copolymer produced. The IR spectra of anethole monomers have absorption characteristics of the methoxy group ($-OCH_3$) at wave number 1035 cm^{-1} and the aromatic ring double bond (C=C) at 1606 cm^{-1}, as well as the absorption of the vinyl group (C=C) at 1696 cm^{-1}, supported by absorption of vinyl groups ($-CH=CH_2$) at wave number 965 cm^{-1}. Whereas in the IR spectra the LMA monomer has absorption characteristics of the –C-O-C-methacrylate group at wave numbers 1296 and 1165 cm^{-1} and the absorption of the carbonyl group from acrylate at wave number 1722 cm^{-1}, besides that the LMA monomer also has an absorption group vinyl (-CH=CH2) at wave number 938 cm^{-1}. The formation of co-poly(Anethole-LMA) was characterized by the disappearance of vinyl group absorption (C=C) in the wavenumbers 1696, 1638, 965, and 938 cm^{-1}. A comparison of functional group absorption on anethole, LMA, and co-poly(Anethole-LMA) monomers are presented in **Table 2**. This indicated that the synthesis of co-poly(Anethole-LMA) had been successfully performed.

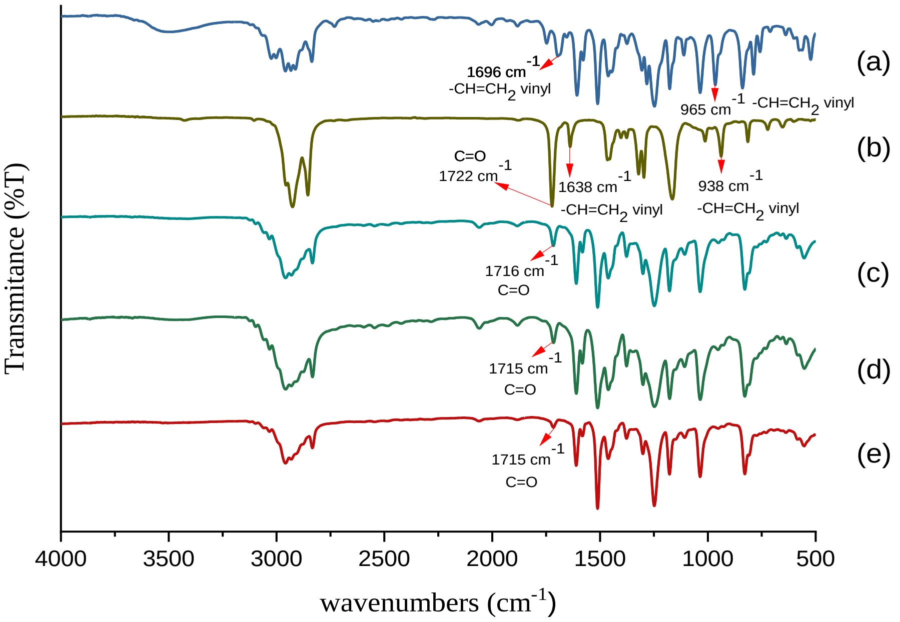

Figure 4. Comparison of FTIR spectra on (**a**) anethole, (**b**) LMA, (**c**) co-poly(anethole-LMA) 2%, (**d**) co-poly(anethole-LMA) 4%, and (**e**) co-poly(anethole-LMA) 6%.

Table 2. Comparison of functional group absorption in anethole, LMA, and co-poly(anethole-LMA).

Functional Group	Wavenumber (cm^{-1})				
	Anethole	LMA	Co-poly (Anethole-LMA) 2%	Co-poly (Anethole-LMA) 4%	Co-poly (Anethole-LMA) 6%
-CH *stretch* (metil)	2836	2854-2956	2833-2957	2833-2957	2833-2958
C=O ester *stretch*	-	1722	1716	1715	1715
C=C *stretch* vinil	1696	1638	-	-	-
C=C *stretch* aromatics	1606	-	1610	1609	1610
C-H *stretch* aromatics	3002	-	-	-	-
C-O-C methacrylate	-	1296; 1165	1248; 1177	1248; 1177	1248; 1177
-CO- methoxy *stretch*	1035	-	1035	1035	1035
=C-H trans anethole	965	-	-	-	-
Vinyl (acrylate) CH=CH$_2$ *bending*	-	938	-	-	-

^{1}H-NMR Analysis

The successful synthesis of co-poly(anethole-LMA) with variations in LMA weight of 2%, 4%, and 6% by weight of anethole can be identified using ^{1}H-NMR. Analysis using ^{1}H-NMR can provide information about the number of protons and the differences in protons in a molecule with the influence of the electronic environment of each proton. 1H-NMR analysis by comparing the spectra of anethole and LMA monomers with the spectra of co-poly synthesized products (anethole-LMA) in various variations. The comparative analysis results of ^{1}H-NMR spectra on anethole, LMA, and co-poly(anethole-LMA) are presented in **Figure 5**.

^{1}H-NMR spectra of co-poly(anethole-LMA) showed almost a loss of the proton signal at chemical shifts (δ_H) 6.37, 6.13, 6.08, and 5.52 ppm which was the proton signal of the vinyl group on anethole and LMA, so it can be estimated that the double bond in the vinyl group between the anethole monomer and LMA had been added to a single bond which was indicated by the appearance of a proton signal on chemical shift (δ_H) 0.9-1.97 ppm. The proton signal in this range indicates that there is a chain extension in the copolymer resulting in overlapping proton signals (Handayani et al., 2019). This indicated that the synthesis of co-poly(anethole-LMA) had been successfully done.

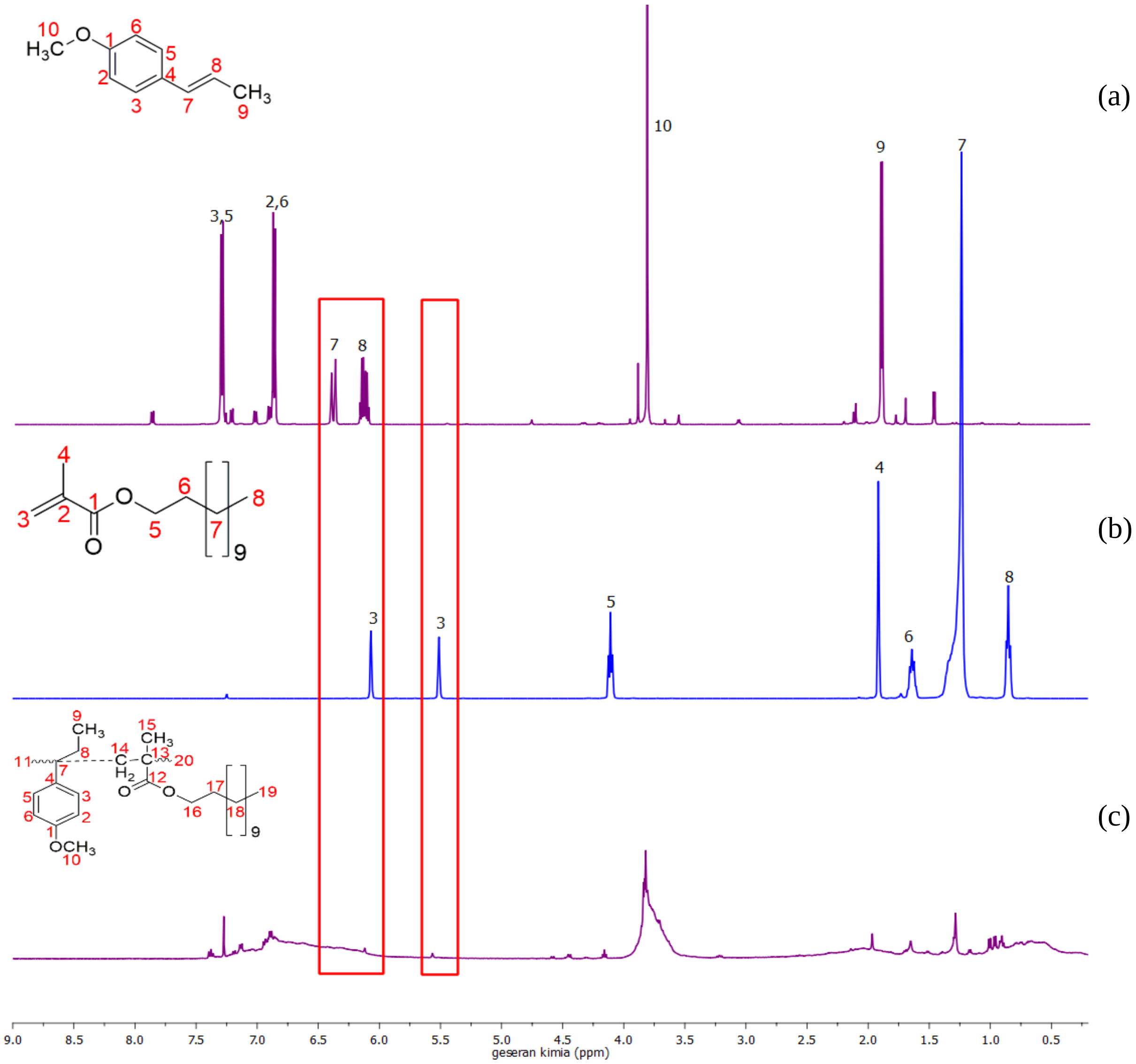

Figure 5. Comparison of ^{1}H-NMR spectra (**a**) anethole, (**b**) LMA, and (**c**) co-poly(anethole-LMA) 2% ($CDCl_3$, 500 MHz).

The M_v of Co-poly(Anethole-LMA)

Relative molecular weight is an essential characteristic that can affect the polymers' properties (Urban et al., 2007). The M_v determination of co-poly(anethole-LMA) was carried out by viscosity measurement using an Ostwald viscometer based on the flow time in chloroform solvent at room temperature. The result of the relative molecular weight (M_v) determination and the degree of polymerization (DP) is presented in **Table 3**.

Table 3 shows that the M_v of co-poly(anethole-LMA) increases with the addition of LMA monomer weight. The increase in the M_v of Co-poly(anethole-LMA) is caused by the addition of the weight of LMA that has a relative molecular weight almost twice the weight of the relative molecular weight of anethole and will increase the active site that the polymer has to bind the added monomer. That is as previously reported in the synthesis of copolymers between eugenol monomers and DVB, where the weight percent of DVB put is in charge of the molecular weight of the polymer. This study revealed that the molecular weight of the polymer was related to the percent of the weight of the DVB used (Prasetya et al., 2020).

The increase in the degree of polymerization shown in **Table 3** is proportional to the increase in the M_v of the polymer. The increase in M_v is proportional to the length of the alkyl chains attached to the polymer. According to Jang and Kim (2000), the alkyl chain distance is an indicator for determining the hydrophobicity properties of a copolymer. Based on the determination of M_v, it shows that co-poly (anethole-LMA) with a variation of 6% contains the longest alkyl chain with the highest hydrophobicity properties in which the higher degree of polymerization, the better the mechanical properties of the polymer (Tomar et al., 2016). The increase in M_v value and degree of polymerization from 4% co-poly(anethole-LMA) to 6% co-poly(anethole-LMA) was not as large as the increase in M_v and degree of polymerization from 2% co-poly(anethole-LMA) to co-poly(anethole-LMA) 4%.

Table 3. Results of the relative molecular weight determination of co-poly(Anethole-LMA)

Copolymer	$[\eta]_i$	M_v (g/mol)	DP
Co-poly(Anethole-LMA) 2%	20.480	32378.62	81
Co-poly(Anethole-LMA) 4%	28.312	50611.05	126
Co-poly(Anethole-LMA) 6%	33.994	65133.79	162

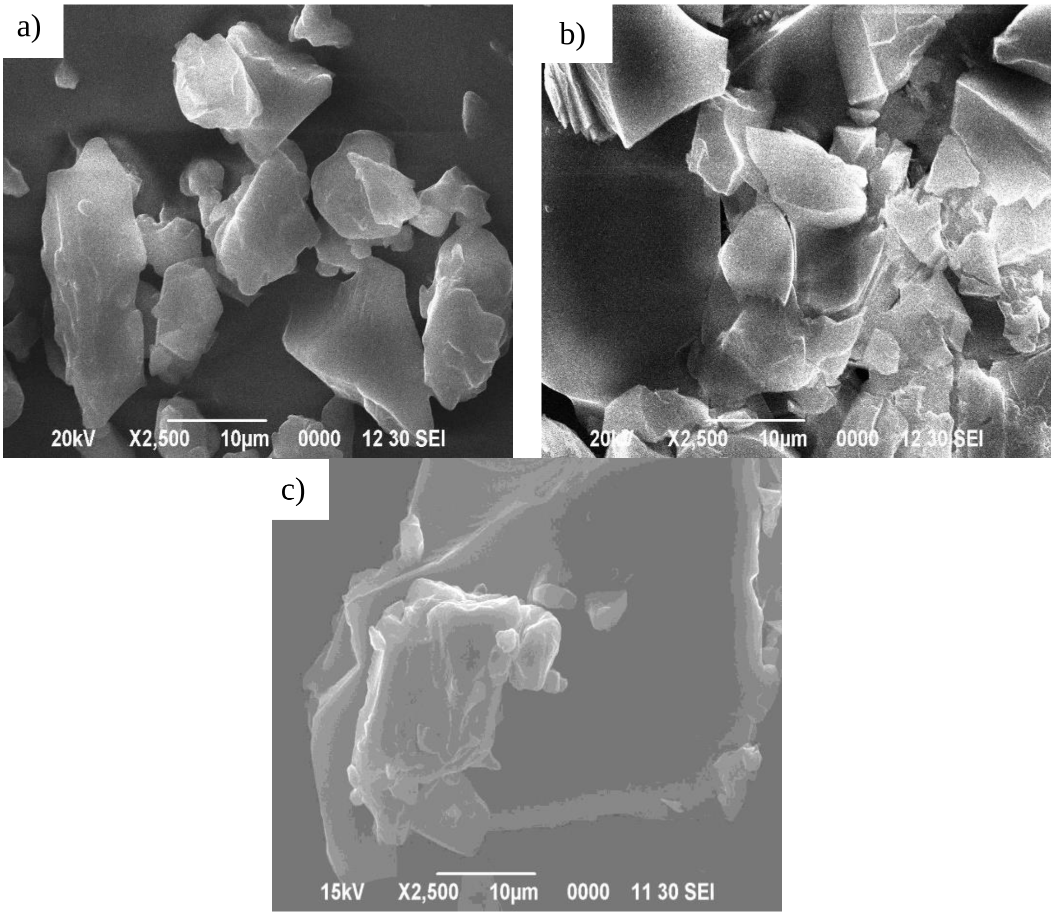

Figure 6. Morphology of (a) co-poly(anethole-LMA) 2%, (b) co-poly(anethole-LMA) 4%, and co-poly(anethole-LMA) 6%.

Table 4. Surface area of co-poly(Anethole-LMA)

Copolymers	Surface Area (m^2/g)	Pore Volume Data (cm^3/g)	Pore Size Data (nm)
Co-poly(Anethole-LMA) 2%	192.20	0.2062	2.146
Co-poly(Anethole-LMA) 4%	98.80	0.096	2.016
Co-poly(Anethole-LMA) 6%	233.80	0.2473	2.115

That is because the copolymer molecules are increasingly abundant by the long LMA chain making the magnitude of the steric factor that occurs in the 6% co-poly(anethole-LMA) also large. These events can affect the rate of propagation in the polymerization process, making it more difficult to attack the carbocation.

Morphological Analysis of Co-poly(Anethole-LMA)

The morphology of 2%, 4%, and 6% co-poly(anethole-LMA) was characterized using SEM as presented in **Figure 6** with a magnification of 2500 times. The morphology of co-poly(anethole-LMA) showed that the higher concentration of the LMA added made the surface distance between particles getting tighter. This suggested that the weight gain of LMA will affect the morphology of copolymers.

Surface Area Analysis of Co-poly(Anethole-LMA)

The analysis of surface area co-poly(anethole-LMA) is shown in **Table 4**. co-poly(anethole-LMA) was included in the type of mesoporous particles because it had a pore size between 2-50 nm. Co-poly(anethole-LMA) 6% had a larger surface area. The higher the weight of the added monomer, the surface area of a polymer will increase. The surface area increases as the pore volume in the material increases, while the pore size is inversely proportional to the surface area and pore volume (Metta et al., 2014). The surface area of Co-poly(anethole-LMA) was 4% lower than co-poly(anethole-LMA) 2%. This can occur because of the possible differences in the distribution of copolymer particles. According to Armini *et al* (2006) the distribution of particles will affect the formation of surfaces on the copolymer.

CONCLUSIONS

Co-poly(anethole-LMA) can be synthesized by cationic polymerization using $BF_3O(C_2H_5)_2$ initiator without solvent media in nitrogen atmosphere conditions. The results obtained are supported by the unique absorption shown by the FTIR spectra at wave numbers 1696, 1638, 965, and 938 cm^{-1} and the almost disappearance of ^1H-NMR spectra at chemical shift (δ_H) 6.4-5.5 ppm that is a signal photons from the vinyl group. M_v co-poly(anethole-LMA) increases with the increasing weight of LMA reacted with anethole. The M_v of co-poly(anethole-LMA) 2%, 4%, and 6% were 32378.62, 50611.05, and 65133.79 g/mol, respectively. The morphology of co-poly(anethole-LMA) showed that the surface distance between particles was getting tighter and the highest surface area in co-poly(anethole-LMA) 6% which was 233.80 m^2/g.

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