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**MECHANICAL AND THERMAL PROPERTIES OF POLYVINYL
CHLORIDE PLASTICIZED WITH NATURAL FATTY ACID
ESTERS**

O. Fenollar, D. Garcia-Sanoguera, L. Sanchez-Nacher, T. Boronat, J. López, R. Balart

Universitat Politècnica de València (UPV), Instituto de Tecnología de Materiales (ITM)

Plaza Ferrándiz y Carbonell sn, 03801, Alcoy (Alicante), SPAIN

Corresponding author: D. Garcia-Sanoguera

Universitat Politècnica de València

Plaza Ferrandiz y Carbonell, sn

03801 Alcoy (Alicante) SPAIN

E-mail: dagarsa@dimmm.upv.es

Tel.: +34 96 652 84 29

Fax: +34 96 652 84 33

Abstract

From an industrial point of view, the use of new nontoxic and biodegradable plasticizers coming from natural resources is an interesting alternative to traditional plasticizers based on phthalates. In this study, two types of epoxidized fatty acids esters (EFAE) with different molecular weight have been used in order to produce flexible PVC with low toxicity. Different amounts of EFAE 201 and 401 have been considered in this study. Mechanical and thermal properties have been determined and morphology of fracture surface has been studied by scanning electron microscopy.

Keywords: polyvinyl chloride; natural plasticizer; mechanical properties; thermal properties,

1.- Introduction

In recent years, there has been an increasing interest in the environmental policies in the polymeric materials sector. This awareness has been presented by consumers, manufacturers and even legislators [1]. This has motivated important lines of work in the development and applications of polymeric materials which are environmentally friendly, low toxicity and easily recyclable [2]. Within this research field, a marked interest has been showed in the improving of the formulations of one of the most widely consumed thermoplastics worldwide as the polyvinyl chloride (PVC), more specifically, the flexible polyvinyl chloride [3, 4]. The use of this thermoplastic is especially sensitive to environmental impact and toxicity problems, since it is widely used in sectors such as plastic toys, childcare, food, packaging, medical-pharmacological... It has to be considered that PVC is obtained from the addition of plasticizers to the vinyl polymer. Some additives and specially plasticizers normally suffer a migration phenomenon to the substances or elements that are in contact [5-8]. So, the contact substances can suffer contamination, and in the worst cases, there is a risk of toxicity and there are health risks to the final consumer [9].

PVC traditional plasticizers based in phthalates are traditionally widely used by the plastic industry. Currently its use is being challenged due to the migration phenomenon, which results in toxicity problems [10, 11]. In order to improve the consumer's health protection and safety, the relevant authorities have developed specific regulations regarding the use of phthalates as PVC plasticizers [12, 13]. The use of the additives: di (2-ethylhexyl) phthalate DEHP, dibutyl phthalate DBP butylbenzyl phthalate BBP di-iso- DINP, DIDP and dioctylftalate DNOP Diisodecyl phthalate, is being limited by the European Parliament (Directive 76/769/EEC, 2007/19/EC, March

2007). This regulation limits the use of the above additives in elements with direct contact with food, toys, childcare, medical devices, cosmetics, pharmacology and biomedicine [14, 15].

Alternative plasticizers have been developed in order to make more flexible the polyvinyl chloride. These plasticizers are characterized by low environmental impact, low migration and low toxicity in applications sensitive to this phenomenon, such as use in childcare items [16, 17]. Plasticizers from natural products are a very interesting alternative as a replacement of phthalates traditionally used in the plastics industry. Interesting research works have been developed successfully for the applicability in the industry of plastic processing [18, 19]. These investigations use citrates, benzoates and carboxylates as alternative plasticizers since they have very similar behavior to PVC flexible with phthalates [20]. In addition, there is an important development on natural plasticizers, with low toxicity and migration for the PVC plasticization [21]. Within this new plasticizers group it should be emphasized the epoxidized derived from soybean oil, linseed oil, castor-oil, sunflower oil and fatty acid esters [22-24]. Inside the range of new plasticizers, the use of epoxidized fatty acid esters acquires some relevance as natural plasticizer. These plasticizers are classified like a secondary kind because they present a double function inside cured plastisol. They act as internal lubricant and as stabilizers too. The epoxidized fatty acids can capture acid groups through catalytic degradation of their epoxide groups, thus the final product is stabilized [25, 26].

A complete study of the curing process is necessary in order to replace traditional plasticizers for the natural ones for optimizing their industrial applications: curing temperature, curing times and optimal amount of plasticizer.

In this study a low toxicity plasticizer as epoxidized fatty acid esters (EFAE), specifically the octyl epoxyoleate has been used, in order to determine the influence of the quantities of natural plasticizer added to PVC on its mechanical and thermal behavior.

The PVC pastes curing process has been carried out under optimized conditions obtained in previous work [25, 27]. The optimum conditions are isothermal curing at 220°C for 10 min, for the same mixing system PVC/epoxidized fatty acid ester. These conditions have been applied to two types of plasticizer: the epoxidized fatty acid esters 201 has lower molecular weight and the epoxidized fatty acid esters 401 with higher molecular weight. Results of previous studies have reported that applying the curing conditions mentioned above, the internal structure of the plastisol acquires an optimal state of curing which is characterized by a completely homogenous morphology that provides an optimal mechanical and thermal response. Therefore, the aforementioned curing parameters are experimentally the most suitable for the production of flexible PVC with these plasticizers of natural origin.

2.- Experimental

2.1.- Materials

The PVC resin that has been used as polymer matrix is Lacovyl PB 1172 H was supplied by Atofina (Atofina UK Ltd., Midlands, United Kingdom) and it has a K value of 67 according to ISO 1628-2, was used as polymer matrix. As plasticizer, two epoxidized fatty acid ester (EFAE, octil epoxyoleate $C_{25}H_{50}O_3$, CAS number 106-84-3) have been used:

- i) 401 with an average molecular weight of 546 g mol^{-1} ,

- ii) 201 with an average molecular weight of 420 g mol^{-1}

Both plasticizers have been supplied by Traquisa (Traquisa S.A., Madrid, Spain). Table 1 shows a summary with the main characteristics of these plasticizers.

Table 1

2.2.-Plastisol preparation

Plastisols were prepared by mixing different amounts of epoxidized fatty acid ester plasticizer (30; 50; 60; 80; 90; and 100 phr) with PVC in a rotative mixer KAPL mod. 5KPMS (KAPL, Michigan, USA) during 10 min at a rotating speed of 3 rpm. After the mixing process, the pastes were placed into vacuum chamber (HEK-GmbH, Lubeck, Germany) under reduced pressure (750 mmHg) for 15 min in order to reduce the volume of air bubbles entrapped in the bulk.

The curing process of the plastisols was performed in a ventilated oven Carbolite mod. 2416CG (Keison Products, Barcelona, Spain) with a maximum temperature of 300 °C. The curing process was carried at isothermal temperature of 220 °C during 10 min. The standardized samples for mechanical characterization were obtained from a plastisol sheet with a die, using a hydraulic press mod. MEGA KCK-15A (Melchor Gabilondo S.A., Vizcaya, Spain).

2.3.- Mechanical characterization

The tensile tests were carried out using a universal tensile test machine ELIB 30 (S.A.E. Ibertest, Madrid, Spain) according ISO 527. A 20 mm min^{-1} crosshead speed was used to obtain the tensile graph with a load cell of 5 kN. All specimens were tested

at room temperature and a minimum of 5 samples were analyzed and average values of tensile strength, elongation at break and elastic modulus were calculated.

2.4.- Microscopic characterization

Morphology analysis of tensile fractures surface of plastisols was carried out by means of a scanning electron microscope JEOL JSM- 6300 (Jeol USA, Peabody) using secondary electrons with an acceleration voltage of 15kV. Samples were covered with a 5–7 nm Au layer in vacuum conditions prior to each measurement.

2.5.- Thermal characterization

The calorimetric analysis was carried out using DSC Mettler-Toledo 821 equipment (Mettler-Toledo Inc., Schwerzenbach, Switzerland). Samples ranging between 4 and 6 mg in weight were used. The heating process was performed from -60 °C to 125 °C at 5 °C·min⁻¹. Tests were performed in an air atmosphere.

The study of thermal stability of blends at high temperatures was carried out with a TGA Mettler-Toledo TGA/SDTA 851 equipment (Mettler-Toledo Inc., Schwerzenbach, Switzerland) programmed with a heating rate of 20°C·min⁻¹ from 50°C temperature to 900°C in an air atmosphere. The specimen crucibles used are made of aluminum oxide with a capacity of 70 µl and ranging between 8 and 10 mg in weight.

3.- Results and discussion.

3.1.- Influence of EFAE amount on mechanical properties

One of the most important aspects in the current study is the optimization of the amounts of plasticizer to be added in the formulation, in order to obtain the polymeric

material with the best performance. The mechanical response of the polymer is an important item to evaluate the suitability of material in addition it is directly affected by plastisol composition. By this way, Figure 1 shows the variation of the tensile strength and elastic modulus as a function of epoxidized fatty acid ester (401 and 201) amount cured at 220°C during 10 min. It can be observed a marked decrease in the tensile strength and elastic modulus with the increase of the amount of plasticizer used for the two fatty acids epoxidized used, 201 and 401.

Figure 1

For low contents of epoxidized fatty acid (30 phr), the tensile strength values are higher. They are about 21 MPa, which are reduced to values close to 10 MPa, when the content increase to 80 phr of either of the two plasticizers.

For content of fatty acid above 80 phr there is a tendency to stabilize the values of tensile strength around 8 MPa. The range of tensile strength values is very similar between these experimental results and those obtained using a traditional plasticizer. The bis(2-ethylhexyl) phthalate (DEHP), which is widely used in industry, can be considered as a reference. The plastisols with DEHP plasticizer show very similar tensile strength values and even for high plasticizer content slightly lower [28].

With regard the elastic modulus, Figure 1 also shows the evolution of the elastic modulus of plastisols as a function of plasticizer content. It can be observed, the same trend that tensile strength. The elastic modulus decreases with the plasticizer content both 201 and 401 plasticizer. The elastic modulus values vary from 12.5 MPa for 30 phr content to 5 MPa for 60 phr content. For higher plasticizer content, the reduction of

elastic modulus is slighter as occurs with the tensile strength, reaching for 80 phr content 2.5 MPa.

Figure 2 shows the variation of elongation at break according to the contents of the two selected plasticizers. There is a rapid increase of the elongation at break for the epoxidized fatty acid 201, it varies from a 175% of elongation at break for a 30 phr content, to 239% for 50 phr content. For epoxidized fatty acid 401 this increase is higher, varying from 148% to 240% of elongation at break. As the plasticizer content increases the elongation at break also does, but the growths are not as pronounced, it presents a tendency to stabilize with either of the epoxidized fatty acid analyzed. In the case of the 201 plasticizer, the elongation at break tends to stabilize at around 255% for variable content between 70 and 100 phr. Whereas in the case of 401, this average value is around 270% for the same range of content. The obtained values of elongation at break are very similar to those presented by the plastisol with DEHP plasticizer [28].

Figure 2

From the results obtained from mechanical tests, it can be concluded that the replacement of phthalic plasticizers by natural non-toxic plasticizers is possible without reducing the mechanical performance of material.

On the other hand, from a structural point of view, the high content in plasticizers, 201 and 401, added to the vinyl resin acts as internal lubricant, which facilitates the sliding of the polymer chains, since it presents less interaction between them. The plasticizer occupies the space between the polymer chains. So, the separation between them is increased, and causes the decline of the attraction polar forces. This

structural change justifies the alteration of the mechanical behavior observed when the amount of epoxidized fatty acid added to PVC is increased, which results in an increase of elongation at break.

3.2.- Influence of EFAE amount on morphology of fracture surface

Figure 3 shows the micrographs of tensile fracture surfaces from tensile test for the samples obtained with low toxicity plastisol with different contents of epoxidized 201 fatty acid.

Regardless of the 201 plasticizer content, a homogeneous and regular aspect has been obtained, without the presence of PVC aggregates which indicates a good state of curing of plastisol [27]. This internal structure corresponds to an optimum cure state that is the responsible of the best levels of mechanical response of plastisol.

Similar fracture morphology has been observed for different 201 plasticizer contents. The obtained surfaces are homogeneous and smooth with little roughness, on which a few small and lighter lines are formed. The amount of these lines increases as plasticizer content increases. In Figure 3a and Figure 3b (30 phr content) can be observed that these lines are straighter and less numerous, and the plastisol presents a 175% elongation at break. The elongation increases up to 239% with 50 phr of plasticizer, which produces larger amounts of more rounded lines at the fracture surface (figure 3c and Figure 3d). For the 90 phr sample (Figure 3e and Figure 3f), it is shown the same phenomenon more pronounced due to the increase of the plastic deformation capacity of the plastisol.

Figure 3

Figure 4 shows the micrographs of the fracture surfaces obtained by tensile for low toxicity plastisol samples with different contents of epoxidized fatty acid 401. The morphologies of the fracture surfaces obtained by tensile test indicate that the curing state of pastes is optimal, as in the previous case.

Figure 4

When different phr of 401 are compared, it is observed that the evolution of the morphologies of the analyzed surfaces (when the amount of added plasticizer to PVC is increased) is very similar to those developed above for the plasticizer 201. The increase of plasticizer 401 content also increases the density of lines on the fracture surface (Figure 4). This density is directly proportional to the increase of the elongation at break quantified in the previous section for these plastisols.

Regardless the type of epoxidized fatty acid used as plasticizer (201-401), the plastic deformation process is favored when the plasticizer content is increased in the low toxicity plastisol formulation, and optimum curing conditions have been applied. As a result of deformation process on the fracture surfaces of the plastisol some grooves or lines have appeared.

3.3.- Influence of EFAE amount on thermal stabilization

Thermal characterization has been carried out in order to complete the study of influence of amount plasticizer on the plastisol. Figure 5 show the DSC calorimetric curves of PVC resin and epoxidized fatty acid ester plasticizers 201 and 401. As it can

be observed, the glass transition temperature (T_g) of PVC is close to 85.0 °C whereas the plasticizers based on epoxidized fatty acid ester structure show an endothermic peak, which indicates the melting point at values close to -8°C and -20 °C for 201 and 401 respectively.

Table 2 resumes the glass transition temperature (T_g) of the plastisol samples with epoxidized fatty acid ester plasticizers 201 and 401, which have been isothermally cured at 220°C during 10 min. The plastisol with 201 plasticizer does not present variation in the T_g values when the plasticizer content is increased, it remains constant at around -14 °C. When the epoxidized fatty acid 401 has been used, the T_g of the plastisol has a slight decrease in a range from -14 to -12 °C.

Figure 5

Table 2

In addition, the calorimetric analysis results do not evidence important differences in vinyl plastisol cured with different amount of 201 and 401 plasticizers. All thermograms obtained by DSC are characterized by a single thermal transition during the heating process. It has not been observed the endothermic peak, which is a characteristic of free plasticizer that has not been absorbed by PVC particles. This fact indicates that there is not free plasticizer and everything has been absorbed by the PVC particles during the gelation process. Therefore the epoxidized fatty acids plasticizers 201 and 401 conduce to a suitable curing state.

Table 3

Table 3 analyses the thermal stability of the plastisol samples based on the content of natural plasticizer. The plastisol suffers thermal degradation during the heating process to high temperature. The inflection point temperature indicates the temperature where thermal degradation is more pronounced. This point increases with the increasing of plasticizer content. This means that there is a significant stabilization of the thermal degradation when the content of natural plasticizer is increased. When the plasticizer 201 is used with 30 phr of plasticizer content, the inflection point is located at 310.69 °C. When the plasticizer content arises up to 60 phr the inflection point is increased to 321°C and it reaches 345.4°C with 100 phr, which means an increase of thermal stability of approximately 11%. In the case of the plasticizer 401, the degradation temperature increases from 304.67 °C up to 324 °C, for contents of 30 phr and 100 phr respectively.

The thermal stabilization effect presented in both epoxidized fatty acid plasticizers analyzed (201 and 401), classifies them as secondary plasticizers. That is, they present a dual action in the vinyl structure: first they plasticize the PVC and also promote a thermal stabilization.

4.- Conclusions

The use of natural plasticizers, such as epoxidized fatty oils is an interesting alternative for the production of low toxicity plastisol with low environmental impact. The curing process can be carried out properly at temperatures around 220°C with curing times around 10 min. In the study of the mechanical properties and thermal

behavior of the plastisol it is shown that content of this type of natural plasticizers can vary depending on the required mechanical properties. For these conditions, the microstructure of plastisol is formed by a completely homogeneous matrix due to the optimal absorption of plasticizer by PVC particles. In addition the 201 and 401 plasticizers promote a noticeably improvement of the thermal stability of plasticized PVC. When optimal conditions of processing are applied, epoxidized fatty acid ester 201 and 401 becomes a suitable choice for use as non-toxic plasticizer for PVC.

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FIGURES

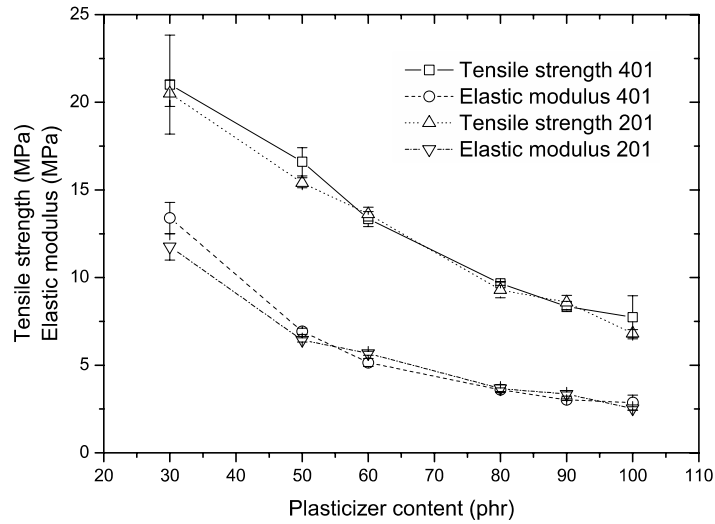


Figure 1. Variation of tensile strength and elastic modulus of vinyl plastisol as a function of epoxidized fatty acid ester (201 and 401) amount, isothermally cured at 220°C during 10 min.

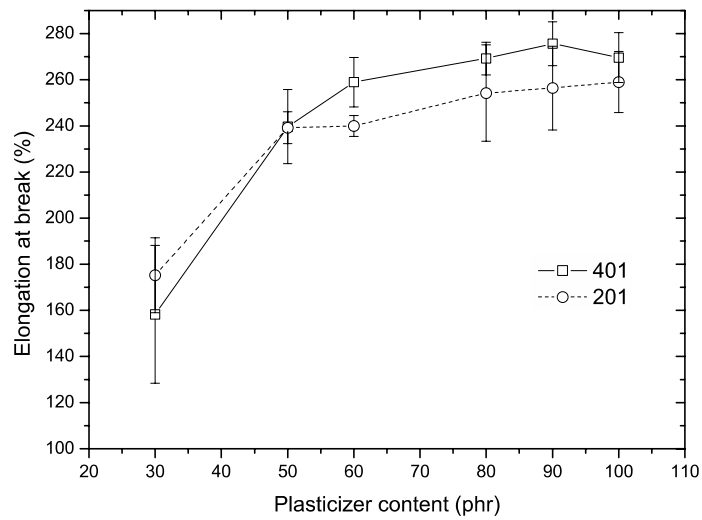


Figure 2. Variation of elongation at break of vinyl plastisol as a function of epoxidized fatty acid ester (201 and 401) amount, isothermally cured at 220°C during 10 min.

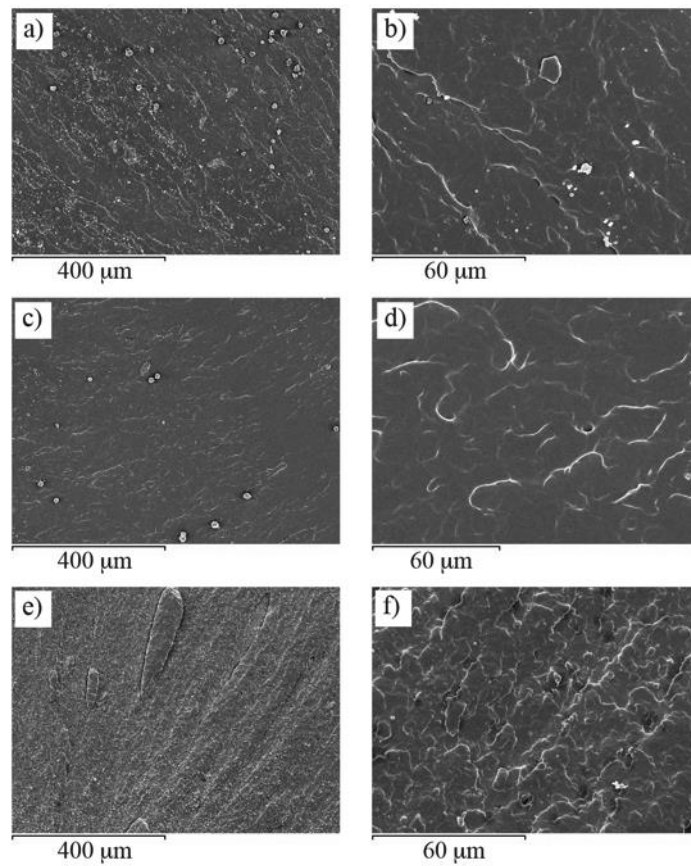


Figure 3. SEM microphotographs of fracture surfaces of vinyl plastisols with different amounts of 201 epoxidized fatty acid ester plasticizer: a) 30 phr x150; b) 30 phr x1000; c) 50 phr x150; d) 50 phr x1000; e) 90 phr x150; f) 90 phr x1000.

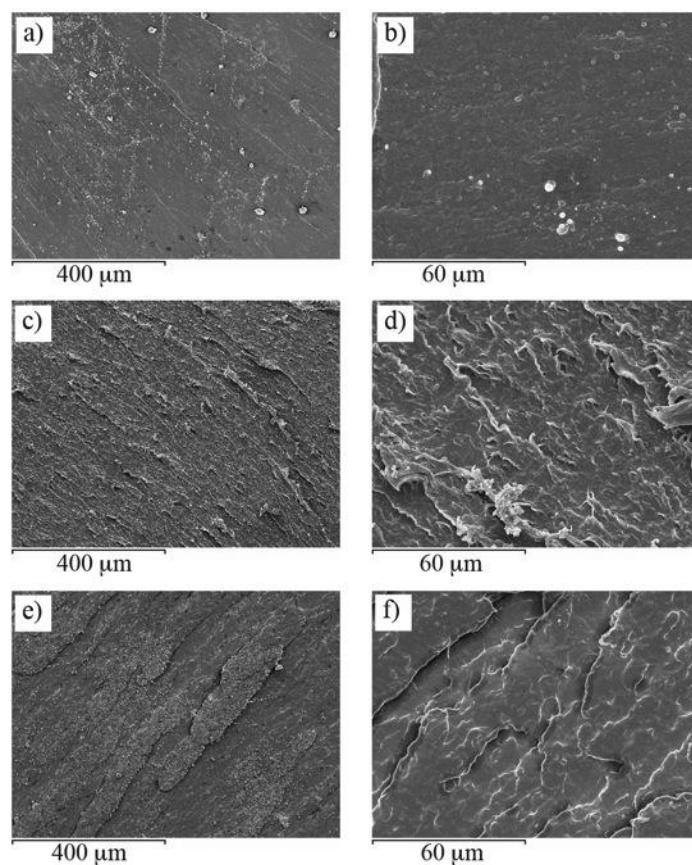


Figure 4. SEM microphotographs of fracture surfaces of vinyl plastisols with different amounts of 401 epoxidized fatty acid ester plasticizer: a) 30 phr x150; b) 30 phr x1000; c) 50 phr x150; d) 50 phr x1000; e) 90 phr x150; f) 90 phr x1000.

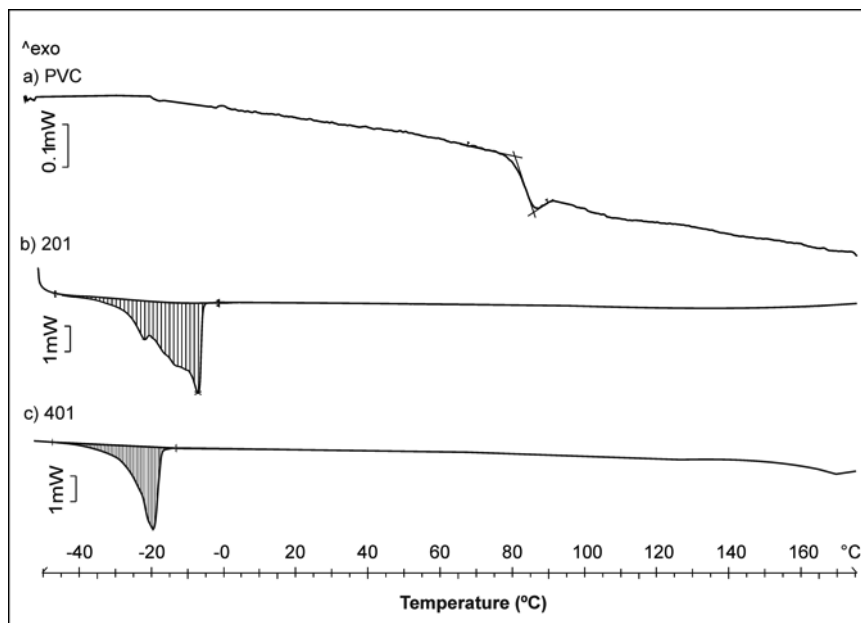


Figure 5. DSC calorimetric curves: a) PVC resin Lacovyl PB 1172 H; b) epoxidized fatty acid ester 201; c) epoxidized fatty acid ester 401.

TABLES

Table 1. Summary of the main characteristics of plasticizers 201 and 401 based on epoxidized fatty acid ester (EFAE) structure.

Properties	EFAE 201	EFAE 401
Density at 20°C, (g cm ⁻³)	0.81 ± 0.01	0.92 ± 0.01
Viscosity at 20°C (cp)	20 - 30	45 - 55
Acid index (mg KOH g ⁻¹)	≤ 1	≤ 1
Refraction index at 16 °C	1.4575 – 1.4580	1.4605 – 1.4610
Iode index	≤ 3	≤ 3
Gardner color	≤ 2	≤ 2
Inflamation point (°C)	>200	>200
Epoxide oxygen (%)	3.1 – 3.3	4.5 – 5
Frezze temperatura (°C)	-20	-18
Water solubility (g L ⁻¹)	Non soluble	Non soluble
Aspect at room temperature	Olious liquid	Olious liquid

Table 2. Tg values obtained by DSC for plastisols with epoxidized fatty acid esters 201 and 401, isothermally cured at 220°C and 10 min.

Plasticizer amount (phr)	EFAE 201 Tg (°C)	EFAE 401 Tg (°C)
50	-14.29°	-14.08°
60	-14.7°	-12.8°
80	-14.35°	-13.67°
90	-14.52°	-11.26°
100	-14.60°	-12.6°

Table 3. Results of thermal degradation obtained by TGA for plastisols with epoxidized fatty acid ester plasticizers 201 and 401, isothermally cured at 220°C and 10 min.

Plasticizer (phr)	EFAE 201			EFAE 401		
	Onset	Endset	Inflection	Onset	Endset	Inflection
30	290.96	333.29	310.69	286.36	330.28	304.67
50	295.13	349.91	319.09	290.35	339.94	313.28
60	293.96	342.34	321.20	289.09	341.10	316.18
80	299.01	348.58	325.97	296.22	345.37	321.86
90	304.51	353.03	331.45	293.25	347.84	323.45
100	291.95	345.39	345.39	292.84	346.89	324.03