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Effect of Plasticizer Percentage on Thermal Properties of Plasticised PVC

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ABSTRACT

The effect of DOP plasticizer percentage on limiting oxygen index and thermal stability of plasticised PVC was investigated in this paper. DOP was added PVC with various quantities (20, 30, and 50) pphr. LOI and thermal stability instruments used for estimating LOI and thermal stability of plasticised PVC as a function to DOP percentage. The results showed that the thermal stability of plasticised PVC was improved by increasing DOP percentage, but in the same time has a reverse behavior where the LOI decreases as the DOP percentage increases.

Introduction

Polyvinyl chloride or PVC is an important and commonly used plastic in many applications, where it can be used as a rigid or plasticized according to the type of application. The properties of PVC relate to its composition whether it is rigid or plasticized, and the methods of production. PVC is characterized by a large molecular weight, where at normal temperatures, a large number of organic compounds have no effect on it. The compounds that attack it are chloro-hydrocarbons and some esters [1-3]. PVC is mainly a rigid material at low temperatures because of high attraction forces between the molecules which results from a short distance between these molecules. These distances begin to expand when the PVC is heated; causing in softness it's structure, but these softness quickly disappears when it cools. So, in order to maintain this softness in the PVC structure even at room temperature, it is added a materials called plasticizers that prevent polymer molecules from approaching to each other, will keep polymer softness due to the large distance between molecules [4]. Fig.1 shown the mechanism of a plasticizer in PVC.

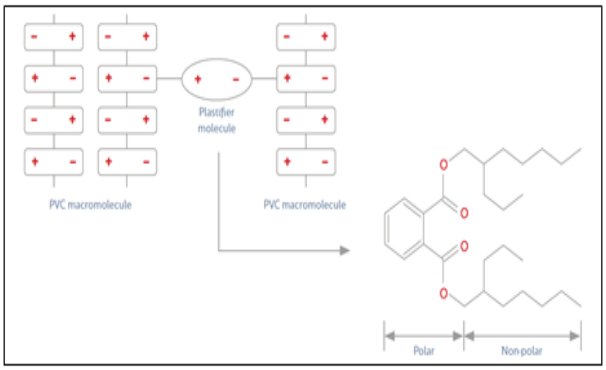

Fig 1. Mechanism of a plasticizer in PVC [5].

Dioctyl phthalate (DOP) is one of the most popular plasticizers in the plastics industry. It has special properties such as high plasticizing ability, low volatility ratio, high UV and water resistance, and good electrical properties.

DOP is widely used in the manufacturing of PVC and is also added to many other resins and rubber [6].

Methodology

a. Materials: the main materials percentages were used in this article are:(1) PVC S-5070 (Ongrovil®) supplied by BorsodChem Zrt., Hungary with specifications shown in Table.1;(2) Dioctyl Phthalate (DOP) as plasticizer supplied by DEZA, a. s. CO.,Valašské Meziříčí, Czech Republic, and the properties of this material shown in Table.2;(3) Newstab-50 as calcium-zinc stabilizer which supplied by Betaquímica CO., Barcelona,Spain,with general specifications illustrated in Table.3; and (4) Wax-E (Licowax® E) as lubricant supplied by Clariant International Ltd,Muttenz Switzerland with specifications and general properties shown in Table.4.

Table 1. Specifications of PVC S-5070 (Ongrovil®) [7].

Parameters		Value
K-value		69-71
Volatile content (max.), %		0.3
Apparent density, kg/m^3		470-530
Particle size distribution, %	above 0.250 mm (max.)	1
	below 0.063 mm (max.)	5
Plasticizer absorption, % (min.)		30
Fish-eyes, pcs/dm^2 (max.)		10
Number of impurities, pcs/9dm^2 (max.)		20
Residual vinyl-chloride content, mg/kg(max.)		1
Specific vol. resistance at 23°C, Ω x cm(min.)		5 ×10^{13}
Glass transition temperature, °C		75-85
Density of melted solid PVC resin, g/cm^3		1.41
Flow temperature, °C		140

Table 2. Specifications of Dioctyl Phthalate (DOP) plasticizer [8]

Parameters	Value
Melting temperature, °C	-50
Boiling temperature, °C	384
Flash point, °C	207
Flame point, °C	233
State at 20 °C	Viscous liquid

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Table 3. Specifications of Newstab-50 stabilizer [9]

Properties	Value		
Viscosity at 20°C, cps	Minimum	Maximum	Average
	220	280	250
Density at 20°C, g/cm³	0.89	0.99	0.94
Ca content, %	0.81-0.83		
Zn content, %	0.64-0.66		
Appearance	Orange liquid		

Table 4. Specifications and general properties of Wax-E lubricant [10]

Characteristics	Value	Test method
Drop point, °C	80	DGF-M-III 3
Flash point, °C	160	DIN 51758
Viscosity, mPa·s	30	DGF-M-III 8 at 100°C
Density, g/cm³	1.01-1,03	DIN 53479 at 20°C
Appearance	light yellow powder	QM-AA-634

b. Samples preparation: The samples prepared from PVC with some additives according to the percentages shown in Table.5. First, the PVC batches prepared by using the laboratory two roll mill type Schwabenthan shown in Fig.3 with processing conditions illustrated in Table.6, these samples made as sheets with 0.4 -0.6 mm in thickness and. Then, the sheets fabricated to the proper shapes by using hydraulic press type burkle at 300 and 20 bar pressure and temperature 175°C. The limiting oxygen index samples were fabricated according to (ISO 4589-2) standard with 80 mm length, 10mm width, and 4mm thickness. (EN ISO 305) standard was used to fabricate thermal stability samples with 200 mm length, 15mm width, and 0.4mm thickness. This test was done with temperature 190°C and 200°C.

Table 5. Composition of batches.

Sample no. / Material, pphr	1	2	3
PVC S-5070	100	100	100
DOP	20	30	50
Newstab 50	1.5	1.5	1.5
Wax-E	0.3	0.3	0.3

Table 6. Condition of rolling process.

Temperature	Time	Rolling speed	
170 ºC	5 min	21 rpm, Front roller	24 rpm, Back roller

c. Thermal tests:

1. Limiting oxygen index test (LOI) was done by the Stanton Redcroft FTA flammability unit found in BorsodChem Zrt (see Fig.2).

2. Thermal stability instrument (Stabilimetr PVC 03) found in BorsodChem Zrt and shown in Fig.3 was used to complete the thermal stability test. This test is finishing within 2 hr, where the sample moves out of the Stabilimetr with velocity 2 mm per minute.

3. Surface SEM-EDX analysis was used for the analysis of plasticised PVC structure. This test was done by using JEOL JSM-IT100 SEM (see Fig.4) found in Faculty of materials science and engineering, University of Miskolc.

Fig 2. Mechanism Limiting oxygen index instrument (BorsodChem Zrt.).

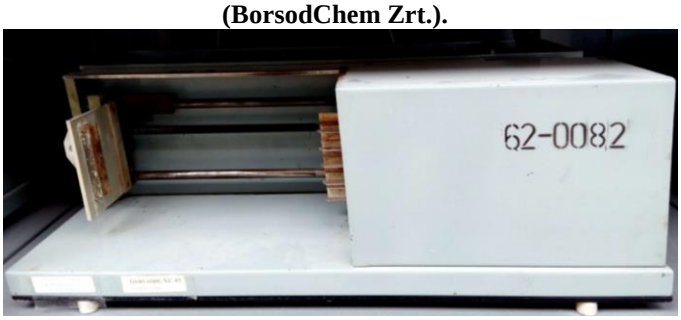

Fig 3. Thermal stability instrument (BorsodChem Zrt.).

Fig 4. JEOL JSM-IT100 SEM analyzer (Faculty of materials science and engineering, University of Miskolc).

Results and discussion

Fig.5 shows the limiting oxygen index (LOI) test vs. DOP content, where the highest LOI is shown by the sample contains 20% DOP, whereas the lowest is shown by sample contains 50% DOP, due to the percentage of oxygen that the burning process needs will increase as the percentage of DOP is decreased. In addition, increasing the percentage of DOP will make PVC more soften and easier to ignite [11,12].

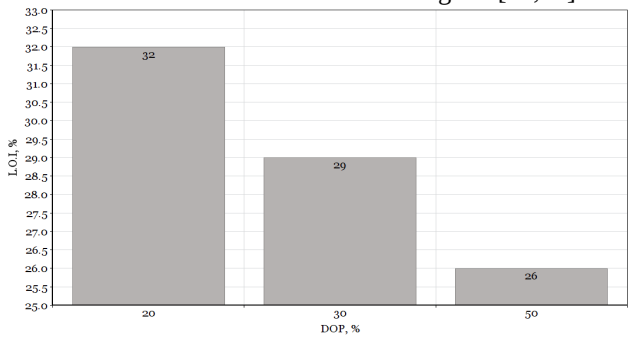

Fig 5. limiting oxygen index of plasticised PVC vs. DOP percentage.

Fig.6 shows the thermal stability of plasticised PVC at 190ºC 200ºC vs. DOP content. From this figure we see that the samples will be a series of gradient colours, where at the beginning of the test, the colour of the sample is light and

transparent because it is not exposed to high temperatures. And when the test continues, the colour starts to change, and the colour deterioration depends on how long the sample stays inside the device, where it will change to yellow, and then to yellowish orange, then yellowish red, then red, then dark red, then brown, then the sample becomes dark. The degree of light or dark depends on the percentage of DOP, where the sample with highest DOP percentage has the best colour stability and it means high thermal stability thermal stability [12,13], and the best results was with temperature 190ºC. The structures of samples was investigated by using scanning electron microscopy (SEM) coupled with energy-dispersive X-ray spectroscopic micro-analyser (EDS) as shown in Figs.7,8,9.

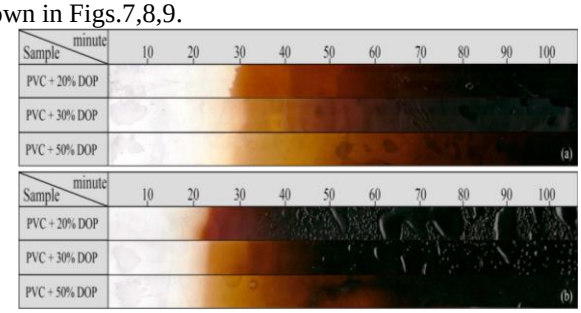

Fig 6. Thermal stability test at temperature 190°C and 200°C respectful.

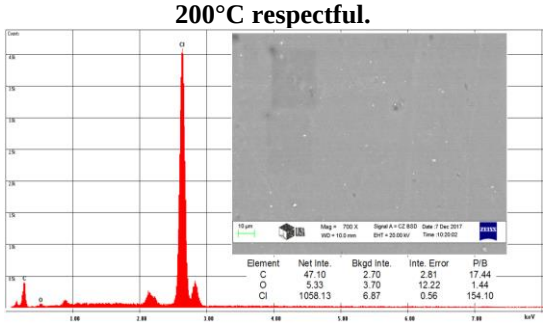

Fig 7. SEM image and energy-dispersive X-ray spectroscopic micro-analyser (EDS) for the sample with 20% DOP.

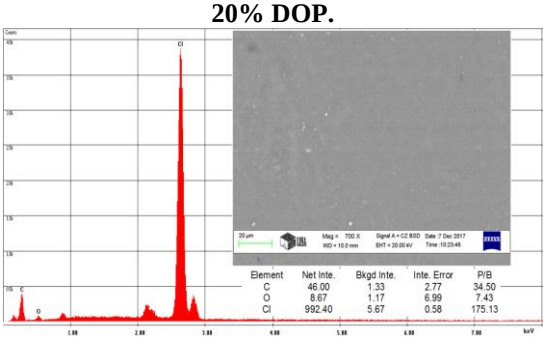

Fig 8. SEM image and energy-dispersive X-ray spectroscopic micro-analyser (EDS) for the sample with 30% DOP.

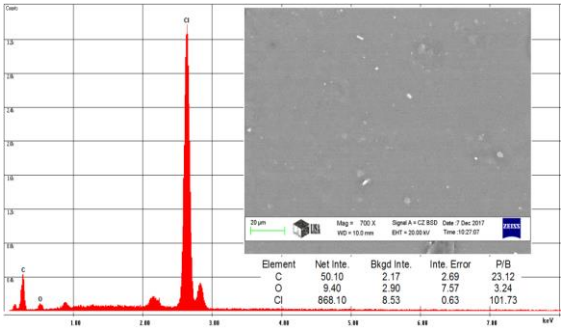

Fig 9. SEM image and energy-dispersive X-ray spectroscopic micro-analyser (EDS) for the sample with 50% DOP.

Conclusions

From the results obtained from thermal results we can concluded that the greater percentage of DOP gives the lower LOI value, because the PVC becomes more soften by increasing the proportion of plasticizer. Conversely, increasing percentage of DOP will improve the thermal stability of the PVC.

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