University of Belgrade, Technical Faculty in Bor

29th International Conference Ecological Truth & Environmental Research

EcoTER'22

Proceedings

Editor
Prof. Dr Snežana Šerbula

21-24 June 2022, Hotel Sunce, Sokobanja, Serbia

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PREFACE

In today's world, the environment has been endangered by the use of outdated technology, fossil fuels and environmental law violations. Therefore, environmental and many other scientists all over the world have been concerned about finding sustainable technology in resolving these issues. That is why environmental research and ecological truth are at the focus of the 29th International Conference Ecological Truth & Environmental Research 2022 (EcoTER'22), which will be held in Sokobanja, Serbia, 21–24 June 2022. On behalf of the Organizing Committee, it is a great honor and pleasure to wish all the participants a warm welcome to the Conference.

We hope to convey the message of the conference, which is that a transformation of attitudes and behavior would bring the necessary changes. This is also an opportunity for the participants who are experts in this field to exchange their experiences, expertise and ideas, and also to consider the possibilities for their collaborative research.

The 29th International Conference Ecological Truth & Environmental Research 2022 is organized by the University of Belgrade, Technical Faculty in Bor, and co-organized by the University of Banja Luka, Faculty of Technology, the University of Montenegro, Faculty of Metallurgy and Technology – Podgorica, the University of Zagreb, Faculty of Metallurgy – Sisak, the University of Pristina, Faculty of Technical Sciences – Kosovska Mitrovica and the Association of Young Researchers, Bor.

These proceedings include 85 papers from the authors coming from the universities, research institutes and industries in 6 countries: Bulgaria, Italia, Albania, Bosnia and Herzegovina, Montenegro and Serbia.

As a part of this year's conference, the 4th Student section – EcoTERS'22 is being held. We appreciate the contribution of the students and their mentors who have also participated in the Conference.

Financial assistance provided by the Ministry of Education, Science and Technological Development of the Republic of Serbia is gratefully acknowledged by the Organizing Committee of the EcoTER'22 conference.

The support of the Platinum donor and their willingness and ability to cooperate have been of great importance for the success of EcoTER'22. The Organizing Committee would like to extend their appreciation and gratitude to the Platinum donor of the Conference for their donation and support.

We appreciate the effort of all the authors who have contributed to these Proceedings. We would also like to express our gratitude to the members of the scientific and organizing committees, reviewers, speakers, chairpersons and all the Conference participants for their support to EcoTER'22. Sincere thanks go to all the people who have contributed to the successful organization of EcoTER'22.

Prof. Snežana Šerbula,

President of the Organizing Committee

RECYCLED POLY(ETHYLENE TEREPHTHALATE) BASED- PLASTICIZER FOR PVC REGRANULATES PRODUCTION

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Abstract

Due to the increasing use and widespread of plastics, poly (ethylene terephthalate) (PET) and poly (vinyl chloride) (PVC) are becoming one of the major threats to the environment. The aim of this paper is obtaining plasticizers from recycled PET, and hot/cold mixing thereof with PVC to produce new materials. Plasticizer was obtained from PET using ethylene glycol (EG) in the presence of catalyst FASCAT 4100, followed by treatment with maleic anhydride and finally 2-ethylhexanol (2-EtHex). The obtained glycolysate and plasticizer, individually or mixtures thereof, were used for the production of polygranulates based on waste PVC in order to obtain an expanded mass of homogenized PVC with glycolysate/plasticizer. The abovementioned procedures and test results for the regranulates and products indicate that the proposed technology offers a solution to the problem of waste PET and PVC through transesterification in order to obtain plasticizer based on PET and EG, called 2-EtHex/MA/PG/PET/EG/MA/2-EtHex used in the processing of PVC waste and in regranulate. The obtained regranulates are further processed by injection molding or extrusion into commercial products such as slippers, footwear, boots, garden hoses, mats, floor mats, etc.

Keywords: PET, PVC, recycling, plasticizers, environment protection

INTRODUCTION

Paying attention to sustainable development is becoming an obligation for socially responsible manufacturers. Recycling as a concept is not enough on its own, so new trends bringing great attention to the circular economy, by increasing the content of recycled material in their final products are being introduced [1]. This puts more pressure on product choice among consumers; the quality and characteristics of the products that are produced must show social responsibility, but also responsibility for the preservation of the environment and resources. Facing these challenges requires improving the quality of recycled granules, speeding up production by expanding the choice of collected industrial waste material. Nowadays, plastic has become a widely used material, due to its low weight and good mechanical properties [2]. PVC and PET are among the most common types of this material [3], because of their possibility of use for food and beverage packaging, as well as in electrical appliances. Due to the diverse application of plastic, it is one of the major polluters of the environment, which is why it is necessary to approach the process of its recycling. Recycling itself involves the processing of waste materials, in order to obtain new, usable materials with appropriate characteristics. Protection of natural resources, as well as lower eco-footprint, represent some of the most important aspects of recycling.

The method applied for recycling of PET was mechanical, in which plastic waste is cut, crushed into granules, flakes or pellets of appropriate quality for production, and then melted to form objects by extrusion [4]. The plastic waste recycled in this way was used to make shoe soles. The degree of injection and extrusion of PVC waste was varied with three different formulations, and the optimal composition of PVC mixtures (extrusion/injection; 70/30) provided good mechanical and physical properties of the sole. By reviewing the literature, in the patent [5], relates to the production of soles for shoes from a composition composed of polyvinyl chloride and organic cellulose. It is necessary to achieve a better quality/price ratio of the produced regranulates and at the same time expand the production capacities. All this will be the result of primary efforts to increase the amount of recycled plastic and reduce the negative impact it has on the environment (when disposed of in landfills without any processing).

The aim of the paper is to achieve a better relationship between the quality and price of produced regranulates and to expand production capacities at the same time. This should be the result of primary efforts to increase the amount of recycled plastic and reduce the negative impact it has on the environment (when disposed of in landfills without any processing).

MATERIALS AND METHODS

Materials

For this study, the following materials were used for the recycling of waste PET: propylene glycol (PG) – Merck, ethylene glycol (EG) – Centrohem. FASCAT 4100 (pmc organometallix) was used as a catalyst for the depolymerization reaction. Ethanol (Sigma-Aldrich), was used to dissolve hydroquinone (Sigma-Aldrich). All chemicals were used without preparation for further purification. Maleic anhydride (MA) - Sigma-Aldrich, was used for esterification of glycolysates based on PET. Other than this, tetrabutyl titanate (Fluka) was also used as a catalyst, and toluene (Sigma-Aldrich) was used in order to remove azeotropic water. Finally, 2–ethyl hexanol (RKS Composites) was used for the synthesis of plasticizers.

Material characterization

Fourier transform infrared (FT–IR) spectroscopy

FT-IR is used for qualitative analysis of functional groups, as well as for structural analysis of various compounds of organic and inorganic origin. FT-IR spectroscopy is applied in the infrared region of electromagnetic radiation with a wave number 4000 - 400 cm^{-1}. The ThermoScientific Nicolet iS10 instrument was used to record the IR spectrum.

Nuclear magnetic resonance

Spectra were recorded at room temperature in deuterated chloroform ($CDCl_3$) or deuterated dimethyl sulfoxide (DMSO-d_6) in 5 mm cuvettes. Chemical shifts are expressed in ppm (d) values relative to TMS (tetramethylsilane) in ^{1}H NMR spectra and solvent residual signal in ^{13}C NMR spectra. NMR spectra were recorded on a Bruker Avance III 500/125 MHz instrument, at 500 MHz when recording ^{1}H NMR and 125 MHz when recording ^{13}C NMR spectra.

Synthesis of 2-EtHex/MA/PG/PET/EG/MA/2-EtHex

The 2-ethyl hexanol terminated plasticizer, based on propylene glycol (PG), was synthesized as follows: 68 g of propylene glycol (0.867 mol) was gradually added to the previously measured PET (167 g, 0.867 mol) in a 1000 ml reactor. The reaction is performed with a gradual increase in temperature to 205-210 °C for 4 hours from the moment all PET is dissolved with addition of 1.5 g FASCAT 4100 as the reaction catalyst. The mixture was gradually cooled to 90 °C, and nitrogen (gas) was introduced due to the inert condition. After that, prepared ethanolic solution of hydroquinone (70 mg of hydroquinone in 0.20 ml of ethanol, 0.02 wt. % was carefully added, followed by 170 g of maleic anhydride (MA, 1.733 mol). The reaction is carried out for 30 minutes at 90 °C and then the temperature is gradually increased to 150 °C.

When the temperature (150 °C) was reached, 2-ethyl hexanol 225.4 g (1.734 mol) was added. A catalyst, tetrabutyl titanate, 1.89 g (0.3% TBT) was added dropwise to support the esterification reaction. Esterification was continued for 1 hour, after which Dean-Stark was adjusted, and toluene was added to remove azeotropic water and the temperature was raised to 210 °C. When 28 ml of water (expected 32.3 ml) was removed from the reaction mixture, the nitrogen inlet was removed and the reactor was vacuumed and distilled until the temperature dropped to 90-100 °C (distillate ceases to separate). The product is poured into a container and used in the processing of waste PVC.

a) *b)*

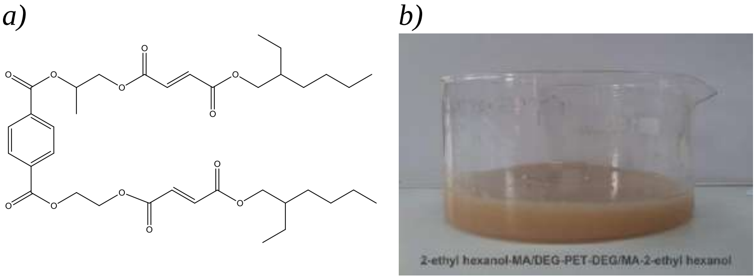

Figure 1 *a) Assumed structure of the obtained plasticizer; b) 2-EtHex/MA/PG/PET/EG/MA/2-EtHex sample*

Production of PVC regranulate based on 2-EtHex/MA/RG/PET/EG/MA/2-EtHex plasticizers obtained from waste PET

Waste PVC is obtained in the form of strips, in bales, which are transported to the mill, where it is prepared for grinding. The waste is grounded through a sieve Ø from 1 mm to 20 mm. The ground waste is mechanically (manually) dosed into a hot mixer. When mixing, the temperature starts at 30 °C. During mixing, 2-EtHex/MA/PG/PET/EG/MA/2-EtHex is added. 10, 50, 100, 150, 200, 250 and 300 g of the plasticizer, depending on the desired hardness, is added per 1000 g of waste PVC. When the temperature reaches 35 °C, the synthesized plasticizer is added to the hot mixer at such a rate that all of the plasticizer is added in up to 5 minutes, mixing with the waste PVC. When the temperature reaches 135 °C, i.e. when the pointer on the ammeter of the upper mixer calms down, this batch is discharged into the cold (lower) mixer. Since the mass from the upper mixer is hot and doughy, after adding to the cold mixer, the mass is converted into a flour form by cooling and bringing to a temperature of 45 °C. It is then drained from the lower mixer and in such a form can be used as a raw material for making the final product - by injection molding. There is no waste during this process, and such a semi-finished product does not have the characteristics of a hazardous product and is ready for further use and production of final products, or can be processed by injection molding to obtain regranulate. The characteristics of the product obtained after formation by extrusion (Figure 2) were tested, and it was determined that the optimal properties of the product are obtained at a mass ratio of PVC/2-EtHex/MA/PG/PET/EG/MA/2-EtHex of 50, ie. 200 g 2-EtHex/MA/PG/PET/EG/MA/2-EtHex per 1000 g PVC, and the material properties obtained in this ratio are shown in Table 1.

a) *b)*

Figure 2 *Structure of regranulate obtained*

RESULTS AND DISCUSSION

FT–IR analysis

On the recorded spectrum, the tensile vibration of the hydroxyl group was observed as a broad band at 3430 cm^{-1}, while the aromatic and vinyl C-H tensile vibration was visible as a shoulder at 3056 cm^{-1}. Asymmetric and symmetric tensile vibrations of the methyl, CH_3 and methylene, CH_2 group, were observed in the range 2977–2873 cm^{-1}, while corresponding bending vibrations occurred at 1454 and 1379 cm^{-1}. The elongation vibration of the ester carbonyl group was assigned a band at 1715 cm^{-1}. The main absorption bands between 1300 and 1100 cm^{-1} are caused by asymmetric and symmetric C-O extensible vibrations found in ester and ether groups.

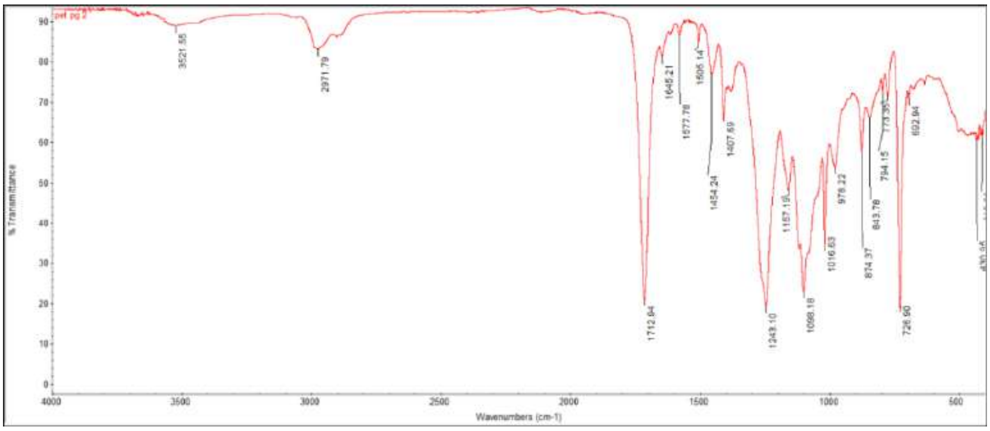

Figure 3 *FTIR spectrum of synthesized* ***2-EtHex/MA/PG/PET/EG/MA/2-EtHex*** *plasticizer*

^{1}H and ^{13}C NMR results

Results of ^{1}H and ^{13}C NMR analysis of **2-EtHex/MA/PG/PET/EG/MA/2-EtHex** plasticizer are:

^{1}H NMR ($CDCl_3$): 0.91–1.33 (m, 9H, 3xCH_3), 3.62–3.69 (m, 4H, 2xCH_2-OH), 4.38–4.71 (m, 2H, -O-(CH_3)CH-CH_2-O-), 4.75–4.77 (m, 2H, 2x-O-(CH_3)CH-CH_2-OH), 4.95 (s, 2H, 2x-CH_2-OH), 5.33–5.38 (m, 2H, 2x-O-(CH_3)CH-CH_2-O-), 6.50–7.06 (m, 4H, 2x-CH=CH), 7.88–8.15 (s, 4H, HPh);

^{13}C NMR ($CDCl_3$):17.51 (3xCH_3), 65.22–72.59 (7xCH_2 and 3xCH), 129.75–133.55 (4xCPh and 2xCH=CH), 165.22–167.81 (6xCOO).

Chemical, physical, electrical and mechanical properties

From the given table it can be seen that regranulates based on synthesized plasticizer have very good characteristics, due to their low ability to adsorb water, as well as resistance to weathering and chemicals.

Table 1 *Characteristics of the product obtained from PVC regranulate in the ratio of 1000g PVC/200g 2-EtHex/MA/RG/PET/EG/MA/2-EtHex*

Chemical properties	
Concentrated acids	Good
Dilute acids	Good
Alcohol	Good
Bases	Good
Aromatic hydrocarbons	
Fats and oils	Good
Halogens	Good
Ketones	Good

Table 1 *continued*

Electrical properties	
Dielectric constant at 1 MHz ASTM D150	4.3
Dielectric strength (125 v mil^{-1}) ASTM D149	370
Dissipation factor at 1kHz ASTM D150	0.05
Volume resistance AST D257 (Ohm cm) at 22.8 ºC 50% RH	5.5×10^{13}
Mechanical properties	
Elongation ASTM D638 (%)	67
Tensile strength ASTM D638	50 MPa
Bending resistance ASTM D790	52.3 MPa
Compressive strength ASTM D695	50.8 MPa
Young's modulus ASTM D638	2.8 GPa
Modulus of elasticity ASTM D790	1.9 GPa
Rockwell hardness R ASTM D785	106
Toughness (J m^{-1})	26
Tearing module (GPa)	2.6
Breaking voltage (MPa)	84
Physical properties	
Density (g cm^{-3}) ASTM D792	1.34
Flammability	Self-extinguishing
Oxygen limiting index (%)	22
Refractive index	1.532
Resistance to UV radiation	Good
Water adsorption (%)	<0.6
Water adsorption - within 24 h (%) ASTM D570	0.74
Coefficient of thermal expansion ($\times10^{-6}$ K^{-1})	46
Lower operating temperature (°C)	-40 to -60
Specific heat (J K^{-1} kg^{-1})	1258
Thermal permeability (W m^{-1} K^{-1})	0.26 at 23 °C

CONCLUSION

This paper aimed to examine the characteristics of plastic materials acquired by mixing PVC with plasticizers obtained by physicomechanical treatment of PET waste. The composition of the gained materials was examined using FTIR spectroscopy and NMR spectroscopy. Based on the presented results, it is possible to conclude that the materials obtained by adding these plasticizers into PVC (extrusion/injection; 70/30) show good mechanical and chemical properties. In addition, due to the acceptable content of acids and bases, as well as good physicochemical and mechanical characteristics, regranulates obtained by mixing the synthesized plasticizer in PVC, will avoid numerous negative effects on the environment. Regranulates with the best characteristics were obtained by mixing 200 g of plasticizer with 1000 g of PVC.

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