Metallurgical and Materials Engineering *Research paper*
Association of Metallurgical Engineers of Serbia AMES *https://doi.org/10.30544/768*

CIRCULAR ECONOMY IMPLEMENTATION IN THE DEVELOPMENT OF FIRE-RETARDANT MATERIALS USED IN CONSTRUCTION, INDUSTRY, AND GENERAL-PURPOSE PRODUCTS

Kaled Mohamed Ben Omran[1], Salem Ibrahim Shwika[1], Marija M. Vuksanović[2], Aleksandar D. Marinković[3], Aleksandar Jovanović[3], Nevena Prlainović[3], Dragana Vasilski[1]*

[1] Faculty of Sustainable Development and Management, University "Union – „Nikola Tesla" Cara Dušana 62-64, 11000 Belgrade, Serbia
[2] University of Belgrade, Department of Chemical Dynamics and Permanent Education, „VINČA" Institute of Nuclear Sciences - National Institute of the Republic of Serbia, Mike Petrovića Alasa 12-14, 11351 Belgrade, Serbia
[3] University of Belgrade, Faculty of Technology and Metallurgy, Karnegijeva 4, 11120 Belgrade, Serbia

Received 29.11.2021
Accepted 13.03.2022

Abstract

During a fire, passive fire protection systems are designed to prevent the spread of flames, smoke, and toxic gases. The new fire-retardant (FR) material, used for passive fire protection, is created by combining copolymers (VC–co–VAc) (Slovinyl KV 173) and PVC K70 with expanded graphite and plasticizers/modifiers such as diisononyl phthalate - DINP, diisononyl terephthalate - DINTP, dioctyl adipate - DOA, as well as plasticizers that are synthesized based on tertiary recycling of waste poly(ethylene terephthalate) (PET), 1-hexadecene, azodicarbonamide (ADC), tri(p-cresyl phosphate), epoxidized soybean oil (ESO) and acrylate emulsion (DH50, Ecrylic, or Flexryl, etc.). The obtained material's morphology was examined using an emission scanning electron microscope (FESEM) field. Tensile testing was used to determine the mechanical properties of the obtained samples, as well as Shore A hardness and toughness using the Charpy impact test. All samples obtained were tested according to non-flammability standards. To conform to the new trend of "green economy," the development of novel eco-friendly FRs with improved thermal and mechanical properties will include careful consideration of environmental protection and sustainable development.

Keywords: PET; flame retardants; mechanical properties; expanding material.

*Corresponding author: Marija M. Vuksanović, *marija.vuksanovic@vin.bg.ac.rs*

Introduction

Because of its excellent mechanical properties, good chemical resistance, superior electrical insulation, and low cost, polyvinyl chloride (PVC) is a thermoplastic that is widely utilized in the industries of furniture, construction materials, textiles, and transportation [1]. Due to the presence of chlorine atoms, neat PVC is a rigid and brittle polymer with a high glass transition temperature and low thermal stability [2]. Plasticizers are commonly used to improve the flexibility, softness, and processability of neat PVC in order to overcome these drawbacks [3, 4]. Diesters of phthalic acid, phthalates, such as dibutyl phthalate and dioctyl phthalate, are commonly used plasticizers for PVC [5]. While phthalate-type plasticizers improve the plasticizing properties of PVC, they are toxic and harmful to the environment [6]. As a result of their non-toxicity, plasticizers derived from renewable resources have received significant scientific attention [7, 8]. However, because these bio-based plasticizers and phthalate-type plasticizers are sensitive to heat or flame, and can spread fire [9], their flame retardancy applications are limited to plasticized PVCs. To improve the flammability of plasticized PVCs, environmentally friendly flame retardant additives must be developed.

Adding flame retardants comprising of halogen, phosphorus, or organic compounds to polymer matrices is one of the classic approaches for imparting flame retardancy to polymers [10, 11]. Flame retardants, such as brominated-flame retardants, primarily used for fire safety, are regulated or banned in many countries since as they burn they release compounds hazardous to both humans and the environment [12].

In recent years, there has been significant progress in the research on flame retardant modification of polyethylene terephthalate (PET), including copolymer phosphorous flame retardant, blend inorganic particles, and post-finishing of PET products [13]. PET is an essential thermoplastic material with outstanding fatigue resistance, chemical resistance, mechanical strength, and processability. It is employed in fibers, membranes, technical plastics, bottles, etc. [14]. When this material is used, a substantial amount of non-biodegradable PET trash is produced. Chemical recycling of PET trash can greatly decrease environmental difficulties caused by incineration or landfill accumulation of this material. It can be reused in the production of new products such as polyester, epoxy, and alkyd resins [15], and plasticizers [16]. Primary (Pre-Consumer Industrial Scrap), secondary (Mechanical Recycling), tertiary (Chemical Recycling), and quaternary (Energy Recovery) waste PET recycling is possible [17]. Recycling waste PET is an economic activity based on the possibility of reuse - either through the use of materials or the recovery of energy. The economic benefits of employing discarded or recycled materials outweigh the expenses that follow, and by minimizing the use of expensive raw resources, productivity also increases [18, 19].

The goal of this work is to create passive expanded fire-retardant materials with improved properties by synthesizing and applying plasticizers from waste poly(ethylene terephthalate) (PET). The aim is to create high-quality products that will help in achieving goals of sustainable development, environmental protection, and the circular economy. The obtained materials' mechanical properties and burning resistance were investigated.

Experimental

Materials

The materials used to prepare the expanded material for passive fire protection are expanded graphite (EG) (Ausbery Carbons, USA), stabilizer (Stabiol CZ 2680 Reagens Deutschland GmbH) and poly(vinyl chloride-*co*-vinyl acetate), trade name Slovinyl KV-173, from FORTISCHEM a. s. Slovakia. Dioctyl adipate (DOA), 1-hexadecene, azodicarbonamide (ADC), tri-(p-cresyl phosphate) (TpKP), propylene glycol (PG), diethylene glycol (DEG), triethylamine, dichloromethane (DCM), maleic anhydride (MA), Hidroquinone (HQ) and 2-ethyl hexanoic acid (2-EtHexCOOH) was supplied by Sigma-Aldrich, Germany. 2-Ethyl hexanol (2-EtHexOH), polyvinyl chloride (PVC) K70, waste PET and epoxidized soybean oil was provided by RKS Kompoziti Čelarevo, Serbia.

Preparation glycolysate from the waste polyethylene terephthalate PET

EG/PET/PG (PETplast1)

PET waste depolymerization was carried out in a four-necked round bottom flask equipped with a condenser, thermometer and inert gas inlet tube. PET glycolysis with PG was performed in the following manner: PG (100 g, 1.32 mol) and Fascat 4100 catalyst (0.50 g, 0.4 mas.%) were added into a reaction flask, and gradual addition of PET (100.0 g, 0.52 mol) was performed to attain complete PET dissolution. The temperature of the reaction was maintained at 180 °C in the course of PET addition, and after it was increased to 210-220 °C and maintained for 8 hours providing efficient mixing. After that period reflux condenser was changed with vacuum distillation apparatus assembled to remove excess of PG. When 57.2 ml of PG was collected the reaction mixture was then cooled to 90 °C before being cast into a storage vessel.

2-EtHexCOOH/EG/PET/DEG/2-EtHexCOOH (PETplast2)

In a four-necked round bottom flask, equipped with a mechanical stirrer, water condenser, thermometer, and a nitrogen inlet tube dried PET (100.0 g, 0.52 mol), DEG (55.12 g, 0.52 mol), and Fascat 4100 (0.62 g, 0.4 percent) were added. The flask was placed in a heated oil bath, and the reaction mixture was kept at 210-220 °C for 6 hours in an inert atmosphere providing efficient mixing. The reaction mixture was cooled to 90 °C after glycolysis, a Dean-Stark water separator was assembled, and 2-ethyl hexanoic acid (0.52 mol) was added to the flask. Xylene (3 wt. % of the reaction mixture) was added to provide water removal forming binary azeotrope, and then the mixture was heated to 210 °C at a rate of 15 °C/h, and held at that temperature until 17 ml of water was separated (stoichiometrically 18.4 ml). The mixture was then cooled to 90 °C before being cast into a storage vessel.

2-EtHexOH/MA/EG/PET/DEG/MA/2-EtHexOH (PETplast3)

In a four-necked round bottom flask, equipped with a mechanical stirrer, water condenser, thermometer, and a nitrogen inlet tube, dried PET (100.0 g, 0.52 mol), DEG (55.12 g, 0.52 mol), and Fascat 4100 (0.62 g, 0.4 percent) was added. The flask was placed in a previously heated oil bath, and the reaction mixture was kept at 210-220 °C for 6 hours in an inert atmosphere providing efficient mixing. The reaction mixture was cooled to 90 °C after glycolysis, a Dean-Stark water separator was assembled, and MA (49.9 g, 0.52 mol) and HQ (0.31 g) were added to the flask. The mixture was heated to 150 °C

with a heating rate of 15 ºC/h and held at a constant temperature for 1.5 h. Afterward, 2-ethyl hexanol (0.52 mol) and xylene (3 wt. %) were added to provide water removal forming binary azeotrope. The mixture was heated to 210 °C at a rate of 15 ºC/h, and held at that temperature until 17 ml of water was separated (stoichiometrically calculated 18.4 ml). The mixture was then cooled to 90 ºC before being cast into a storage vessel.

Preparation of expanding fire-retardant material

The hot mixer (10 l) is filled with VC–*co*–VAc copolymer (Slovinyl KV 173) (30 wt. %) and plasticizers DOA (15 wt. %), DINP (10 wt. %), and ADC (0.4 wt. %), TpkP (10 wt. %), ESU (3 wt. %). After stirring for t = 2 hours at T = 110 °C and 3200 rpm, expanding agents are added: expanded graphite (EG) 32 wt.%, 0.2 wt.% 1-hexadecene, 2.5 wt.% of poly(vinylacetate) emulsion (DH50). This sample is designated as Sample 1. In the same way as Sample 1, a sample was made with PVC K-70 which was marked as Sample 2. Materials were prepared using VC-*co*-VAc copolymer (Slovinyl KV 173) (30 wt. %) and PET plasticizers, where instead of DOA, glycolysates PETPlast1 (15 wt. %) (Sample 3), PETPlast2 (15 wt. %) (Sample 4) and PETPlast3 (15 wt. %) (Sample 5). The materials were made with PVC K-70 and glycolysates PETPlast1 (15 wt.%), and were named Sample 6 and with glycolysate PETPlast2 (15 wt.%) (Sample 7), PETPlast3 (15 wt. %) (Sample 8). After mixing, the mixture is placed in molds to produce fire strips of varying lengths. The samples ranged in thickness from 4 to 6 mm, width from 70 to 400 mm, and length from 240 to 500 mm.

Material characterization

The cross-section of obtained material was examined using a Mira3 Tescan field emission scanning electron microscope (FE-SEM) set to 20 kV.

The Shore A durometer hardness of the sample was determined using ASTM D2240 [20]. Each hardness value is an average of eight measurements.

An ELEMENTAR Vario EL III CHNS/O analyzer was used for elemental analysis. Bruker Avance III 500 spectrometer was used to record 1H and ^{13}C NMR spectra in DMSO-d_6. Relative to tetramethylsilane, chemical shifts are given (TMS).

The FTIR spectra of the acquired sample were collected using a Thermo Scientific Nicolet 6700 spectrometer in attenuated total reflectance (ATR) mode with a single bounce 45 °F Golden Gate ATR accessory with a diamond crystal and an electrically cooled DTGS detector. The spectra were ATR corrected co-additions of 64 scans at a resolution of 4 cm^{-1}. Spectra in the wavelength range of 2.5 m to 20 m were recorded using the Nicolet 6700 FTIR spectrometer.

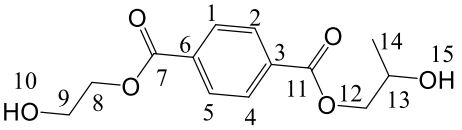
An INSTRON1332 servo-hydraulic testing machine (Instron Ltd., USA) with FASTtrack 8800 control electronics was used to measure the sample's tensile strength. The tensile strength is 5 millimeters per minute. All of the samples were the same length, and three of them were tested, with the mean value calculated and displayed.

The flammability resistance of materials was tested in accordance with the non-combustibility standards AS/NZS 1530.3: 1999 and AS 1530.4-2005.

Results

The synthesized glycolyzate was characterized using NMR, FTIR and volumetric methods. For NMR analysis all products were dissolved in DCM, filtered and after drying and solvent removal used for NMR spectra recording.

The success of the synthesis of *PETPlast1 (PET:PG = 1:1)* plasticizer was proved based on the results of NMR characterization with a dominance of the following compound (evaluated according to NMR results):



^{1}H NMR (400 MHz, DMSO-d_6, δ/ppm): 0.97 – 1.15 (m, 3H, C(14)H_3), 3.56 – 3.74 (m, 2H, C(9)H_2), 4.30 – 4.97 (m, 5H, C(12)H_2, C(13)H and C(8)H_2), 7.90-8.15 (m, 4H, C(1)H, C(2)H, C(4)H and C(5)H);

^{13}C NMR (125 MHz, DMSO–d_6, δ/ppm): 30.1 (C_{14}), 60.1 (C_9), 65.4 (C_{13}), 66.8 (C_8), 129,7 (C_1, C_2, C_4 and C_5), 134.2 (C_3 and C_6), 165.8 (C_7 and C_{11}).

Elemental Analysis (calculated, %): C, 58.20; H, 6.01; O, 35.78

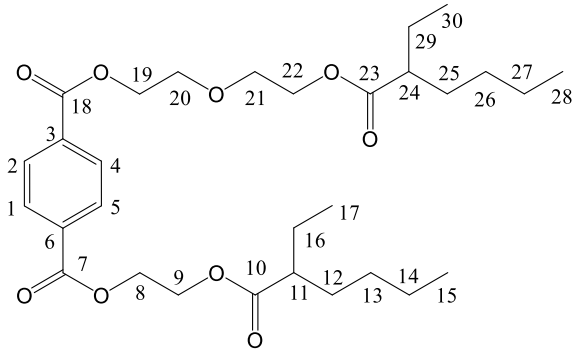
Elemental Analysis (experimental, %): C, 59.40; H, 6.15; O, 34.45

Results of the ^{1}H and ^{13}C NMR analysis of the plasticizer *PETPlast2* are: ^{1}H NMR (400 MHz, DMSO-d_6, δ/ppm): 0.75-0.95 (m, 12H, C(15)H_3, C(17)H_3, C(28)H_3 and C(30)H_3), 1.05-1.48 (m, 8H, C(13)H_2, C(14)H_2, C(26)H_2 and C(27)H_2), 1.52-1.82 (m, 8H, C(12)H_2, C(16)H_2, C(25)H_2 and C(29)H_2), 2.19-2.41 (m, 2H, C(11)H and C(24)H), 3.42-4.75 (m, 12H, C(8)H_2, C(9)H_2, C(19)H_2, C(20)H_2, C(21)H_2 and C(22)H_2), 7.82-8.10 (m, 4H, Ph).

^{13}C NMR (125 MHz, DMSO-d_6, δ/ppm): 12.0 (C_{17} and C_{30}), 13.8 (C_{15} and C_{28}), 22.8-32.1 (C_{12}, C_{13}, C_{14}, C_{16}, C_{25}, C_{26}, C_{27}, C_{29}), 47.1 (C_{11}, C_{24}), 58.8-63.0 (C_8 and C_9), 65.3-69.2 (C_{19}, C_{20}, C_{21}, C_{22}), 129.8-134.8 (Ph), 165.7-175.9 (C_7, C_{10}, C_{18}, C_{23}).

Elemental Analysis (calculated, %): C, 65.43; H, 8.42; O, 26.15

Elemental Analysis (experimental, %): C, 66.21; H, 8.72; O, 25.07

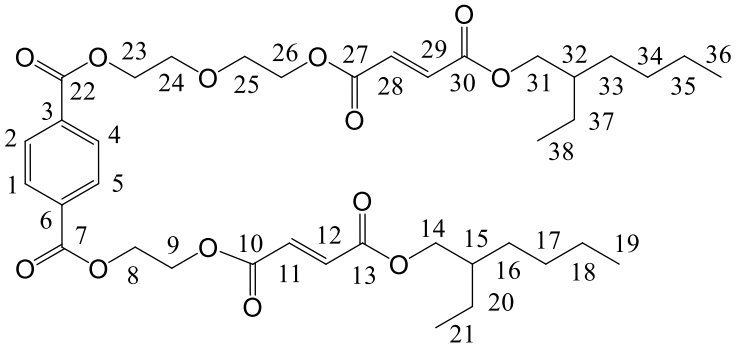


Results of the ^{1}H and ^{13}C NMR analysis of the *PETPlast3* plasticizer based on product of PET glycolysis with DEG are: ^{1}H NMR (400 MHz, DMSO-d_6, δ/ppm): 0.82-1.07 (m, 12H, C(19)H_3, C(21)H_3, C(36)H_3 and C(38)H_3), 1.24-1.53 (m, 16H, C(16)H_2, C(17)H_2, C(18)H_2, C(20)H_2, C(33)H_2, C(34)H_2, C(35)H_2, C(37)H_2), 2.25 (s, 2H, C(15)H and C(32)H), 3.41-3.88 (m, 8H, C(23)H_2, C(24)H_2, C(25)H_2 and C(26)H_2), 4.33-4.98 (m, 8H, C(8)H_2, C(9)H_2), C(14)H_2, and C(31)H_2), 6.46-7.12 (m, 4H, C(11)H, C(12)H, C(28)H and C(29)H), 7.99-8.10 (m, 4H, Ph);

^{13}C NMR (125 MHz, DMSO-d_6, δ/ppm): 11.7 (C_{21} and C_{38}), 14.2 (C_{19} and C_{36}), 23.5-30.2 (C_{16}, C_{17}, C_{18}, C_{20}, C_{33}, C_{34}, C_{35}, C_{37}), 39.8 (C_{15} and C_{32}), 61.0-63.8 (C_8 and C_9), 64.3-69.2 (C_{14}, C_{23}, C_{24}, C_{25}, C_{26} and C_{31}), 129.8-133.9 (C_1, C_2, C_3, C_4, C_5, C_6, C_{11}, C_{12}, C_{28} and C_{29}), 165.7-167.3 (C_7, C_{13}, C_{22} and C_{30}).

Elemental Analysis (theoretical, %): C, 63.49; H, 7.57; O, 28.93

Elemental Analysis (experimental, %): C, 64.18; H, 7.66; O, 28.16



The results obtained from NMR and elemental analysis indicate product heterogeneity of the PET based plasticizers. The morphology and porosity of the cross-section of the obtained fire-retardant material are shown in Figure 1 at different magnifications.

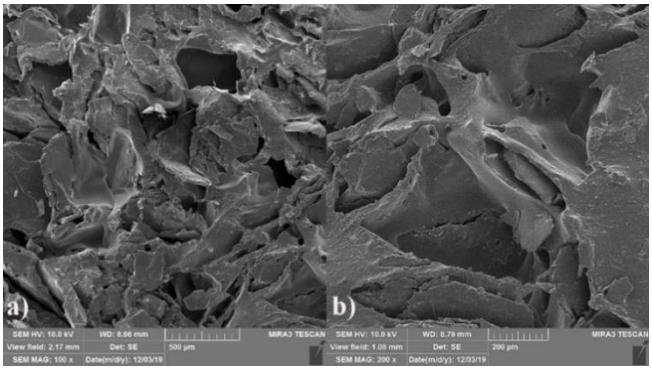


Fig. 1. The morphology of the cross-section of the obtained FR material (Sample 3) at different magnification.

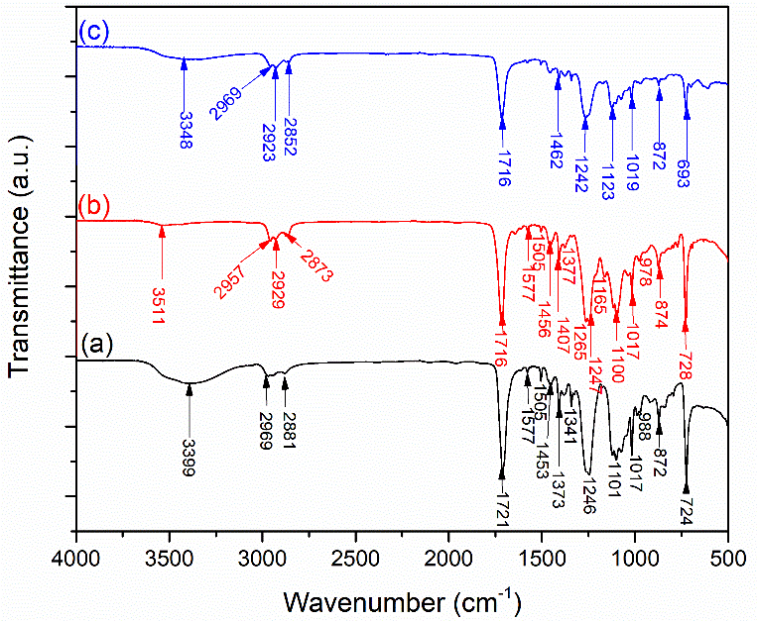


Fig. 2. FTIR spectrum of the obtained material with a) PETPlast1, b) PETPlast2 plasticizers and c) FR Sample 3.

The results of the hardness and specific density of the obtained materials are shown in Table 1.

Table 1. Results of hardness and specific density of obtained fire-retardant materials.

	Hardness (Shore A)	Specific density, g/cm^3	Expansion coefficient, 10^{-5}/K
Sample 1	47 ± 1.4	0.887	10
Sample 2	58 ± 2.4	0.889	7
Sample 3	56 ± 1.3	0.956	7
Sample 4	52 ± 1.5	0.962	8
Sample 5	50 ± 1.9	0.966	9
Sample 6	63 ± 2.1	0.979	5
Sample 7	60 ± 2.7	1.023	3
Sample 8	59 ± 2.5	1.050	4

The results of testing the mechanical properties, specific weight and resistance to the burning of the obtained materials are shown in Table 2.

Table 2. Results of mechanical properties of obtained fire-retardant materials.

	σ, MPa	ε, %	Absorbed energy determined by Charpy method, W (kJ/m^2)
Sample 1	49 ± 1.5	9.2 ± 0.01	157
Sample 2	46 ± 1.7	6.2 ± 0.03	153
Sample 3	45 ± 2.9	7.8 ± 0.03	149
Sample 4	42 ± 2.5	8.2 ± 0.06	146
Sample 5	40 ± 1.6	9.6 ± 0.07	131
Sample 6	38 ± 1.7	5.4 ± 0.06	127
Sample 7	37 ± 2.3	6.8 ± 0.07	120
Sample 8	34 ± 2.1	7.2 ± 0.08	123

Discussion

On the cross-section of the obtained material recorded on the SEM, it can be seen that the material is mainly homogeneous with low porosity. Analysis of SEM images using Image Pro Plus software showed that the porosity amounts to 2.19%.

The FTIR spectrum of the obtained material with glycolysate from PET, *PETPlast1* and *PETPlast2*, and FR Sample 3 are shown in Figure 2. The –OH group stretching vibration, in the FTIR spectra of *PETPlast1* and *PETPlast2*, appeared as a broad absorption band at 3399 and low intensity band at 3511 cm^{-1}, respectively. The methylene and methyl groups stretching vibration was found in the region 2957-2852 cm^{-1}. The strong absorption bands at 1721 and 1716 cm^{-1}, found for *PETPlast1* and *PETPlast2*, respectively, indicate the presence of C=O ester group vibration. Also, C-O-C stretching vibration, observed at 1100 and 1101 cm^{-1}, and aromatic ring out-of-plane deformation vibration at 724 and 728 cm^{-1}, respectively [21], confirmed the structure of the synthesized plasticizer. The stretching vibrations of C–O bond in the ester group were assigned to the absorption peak at 1246 and 1247 cm^{-1} [22]. The bands in the region 872-724 cm^{-1} was ascribed to the C=C stretching (skeletal) vibration of the p-substituted benzene ring [23].

The presence of a hydroxyl (-OH) group was indicated by the broad absorption band at 3348 cm^{-1}. The –CH vibrations stretching (2969 cm^{-1}), CH_2 asymmetric stretching (2923 and 2852 cm^{-1}), CH_2 wagging (1462 and 1242 cm^{-1}), and ester carbonyl stretching vibration appeared at 1716 cm^{-1}. The stretching vibration of C–Cl bond, observed in PVC [24,25], was found at 693 cm^{-1}.

The Shore hardness results are a useful measure of the relative resistance to indentation of different polymer grades. Despite the fact that these data cannot be used to predict other polymer qualities like strength, they are frequently employed as a mechanical performance metric [26]. According to the results shown in Table 1, it can be seen that the addition of PET plasticizer increased the hardness of the obtained material. The hardness of the material with VC-*co*-VAc copolymer and PET/PG plasticizer increased by 19.1 %. The addition of *PETPlast2* plasticizer increased the hardness by 10.6 % but the addition of *PETPlast3* increased by 6.38 %. The hardness of the material containing PVC K-70 copolymer is lower, and the addition of *PETPlast1*, *PETPlast2*, and *PETPlast2* plasticizers increased the Shore hardness from 1.72 % to 8.62 %, respectively. Furthermore, the addition of PET plasticizers raised the specific density and expansion coefficient of the given materials.

Table 2 shows that Sample 1 has the highest tensile strength. The addition of plasticizer from PET and other flame retardants reduced material tensile strengths [27]. Tensile strength is reduced by 8.16 % when *PETPlast1* plasticizer is added to VC–co–VAc copolymer materials, by 14.3 % *PETPlast2*, and 18.4% sa *PETPlast3* plasticizers. PVC K-70 had a similar reduction in tensile strength. The addition of *PETPlast1*, *PETPlast2*, and *PETPlast3* plasticizers results in 17.4 %, 19.6 %, and 26.1 % reductions, respectively. Materials containing *PETPlast1*, *PETPlast2*, and *PETPlast3* plasticizers, on the other hand, have higher elongation values than materials containing commercial plasticizers.

The Charpy impact strength test is a standard ASTM test for determining toughness. This is due to the fact that an impact test indicates a material's toughness, or impact strength, which is the ability of the material to absorb energy during plastic deformation. A tough material is one that has high impact resistance. Toughness is the result of combining strength and ductility. To be tough, a material must have sufficient strength and ductility to withstand cracking and deformation under impact loading. The toughness of the material with VC–co–VAc copolymer and the addition of *PETPalst1*, *PETPlast2*, and *PETPlast3* plasticizers decreased from 2.55 % to 7.1 % in the Charpy impact test. This reduction ranges from 3.05 to 8.4 % for FR containing PVC K-70.

All obtained samples were tested in accordance with non-flammability standards (AS/NZS 1530.3: 1999 and AS 1530.4-2005) [28]. When the experiment was terminated, the samples behaved according to the prescribed standards, halting the flow of air and the spread of fire for 3 hours. All of the samples presented meet the requirements of the standard.

Conclusion

Plasticizers derived from waste PET were synthesized and used to create expanding fire-retardant materials with improved thermal stability in this study. The process of preparing firefighting materials is divided into two stages.

The first phase involves the mixing of copolymers (VC–*co*–VAc) (Slovinyl KV 173) and PVC K70 with expanded graphite and plasticizers/modifiers such as diisononyl phthalate - DINP, dioctyl adipate - DOA, as well as plasticizers synthesized based on tertiary recycling of PET such as: *PETPlast1*, *PETPlast2*, and *PETPlast3*, 1-hexadecene, azodicarbonamide (ADC), tri(p-cresyl phosphate), epoxidized soybean oil (ESO) and acrylatepoly (vinylacetate) emulsion DH50. The second stage involves molding the obtained mixture to create samples of various sizes that are then tested.

According to the results, the addition of plasticizers derived from waste PET increased the hardness of fire-retardant materials by 1.72 % to 19.1 % when compared to materials containing commercial plasticizers. The addition of PET plasticizers and other flame retardants decreased the tensile strength of the material from 8.16 % to 26.14 %. According to Charpy, the toughness of the material is also reduced from 2.55 % to 8.4 %. The addition of PET plasticizers raised the specific density and expansion coefficient of the given materials. All obtained samples were tested in accordance with the non-flammability standards and met the requirements of the standard. If an economically viable recycling process is implemented, the implementation of a circular economy could turn environmental challenges into opportunities for development.

Acknowledgment

This work was financially supported by The Ministry of Education, Science and Technological Development of the Republic of Serbia (Contracts No.451-03-68/2021-14/200135, 451-03-9/2021-14/200017) and Innovation Fund of the Republic of Serbia and Smart Fire Blocks Innovation (Project "Smart Fire Protection – 1297") funded the research. Authors thanks to Dr Nataša Tomić, project leader, for coordination of the activities in the course of project development.

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