

## SUSTAINABLE DEVELOPMENT IN WPCBs TREATMENT FOR PRODUCTION OF BITUMINOUS WATERPROOFING MATERIALS

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### Abstract

Due to the rapidly increasing use of electrical devices, e-waste became one of the major threats to the environment. Waste printed circuit boards (WPCBs) are an integral part of electrical devices, thus the interest in their recycling is growing. Recycling of WPCBs can be performed by applying hydrometallurgical or pyrometallurgical processes, as well as vacuum pyrolysis. The main advantages of vacuum pyrolysis are eco-friendliness, economic viability and efficiency in extracting present noble metals, while pyrolysis oil occurs as a by-product. Pyrolysis oil poses a tremendous threat to the environment, due to the possibility of its spillage into water, as well as a potential release into the air and soil. Because of this, the pyrolysis oil was subjected to chemical treatment with different reagents, such as sodium hydroxide, calcium oxide, sulfuric acid and maleic anhydride, with the intent to solidify the material and remove unpleasant odors. Incorporation of the solidified materials into bitumen, at 2.5, 5, 7.5, and 10 wt.% addition, was performed to obtain waterproofing materials. The obtained solidified and bituminous water-resistant materials were subjected to various test methods: FTIR spectroscopy, physicochemical and mechanical properties of bitumen, etc. which showed that all the tested characteristics are in accordance with values prescribed by the current standard. Toxicity Leaching Procedure (TCLP) confirmed non-hazardous characteristics of the obtained materials, except for the ones with added solidified pyrolysis oil, with Sodium hydroxide, that showed higher leaching of the phenol constituent.

**Keywords:** WPCB; bitumen; recycling; metals; environmental; pyrolysis.

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## **Introduction**

Printed circuit boards (PCBs) are an integral part of almost all types of electrical devices [1]. Nowadays, electronic equipment such as mobile phones, computers, chargers, laptops, etc. are used daily, and because of their shortened lifespan, the amount of electrical waste is significantly increasing [2]. Waste electrical and electronic equipment (WEEE) is one of the fastest growing types of waste in the world [3], with an estimated amount of around 40 – 50 million tonnes per year. The heterogeneous composition of waste printed circuit boards (WPBCs) is characterized by the presence of a large number of organic matter (PAHs, fenantren, naphthalene, etc.), metals (copper (Cu), silver (Ag), gold (Au), palladium (Pd), tin (Sn), iron (Fe), nickel (Ni), etc.), as well as fiberglass embedded in resins. That's why it is justified to approach the recycling process in order to recover metals and phenolic-based resins. Recycling of this kind of waste is very important because of the hazardous components present in WPCBs that can be released into the environment during some processes, such as thermal degradation, as a result of which they have significant impacts on public health, through the pollution of drinking water, agriculture plants and air [4]. Recycling of WPCBs can either be done hydrometallurgically or pyrometallurgically. Hydrometallurgical processes are becoming more popular due to the low capital cost and low environmental impact, and they mainly consist of various combinations of leaching, purification, and metal recovery operations [5, 6, 7]. On the other hand, pyrometallurgical methods, as a mature technology, are more predictable and exact, and therefore easier to be controlled. Yet, during the high-temperature processing, bromides, chlorides, and fluorides which are contained in the polymer fraction may cause highly hazardous gaseous emissions [1]. Some of the downsides of this process in comparison to the hydrometallurgical are high costs of recovery and the possibility of environmental pollution by the generated waste. The products of pyrolysis are organic compounds that are degraded to lower molecular weight and can be used as fuel or chemical sources, but the fiberglass from the resin remains mostly intact, and can therefore be recycled into composite materials.

In this paper, the vacuum pyrolysis method was applied to obtain oil from waste printed circuit boards, due to its numerous advantages [8]. Taking this into consideration, vacuum pyrolysis is considered to be more efficient, environmentally friendly, and cost-effective than the aforementioned metallurgical processes. In particular: (i) vacuum pyrolysis process allows for the low-temperature decomposition of the bonding components, epoxy or phenolic resin, to low-weight gaseous hydrocarbons (5-10%), pyrolysis oil (15-20%) and carbonaceous solid residue (5%); (ii) due to the vacuum, the volume of gas, that needs to be treated, can be reduced to the maximum extent, emitting exclusively low-weight hydrocarbons which could be stored or directly utilized; (iii) due to constant vacuum, the organic vapor leaves the reactor quickly, reducing the occurrence and intensity of secondary reactions; (iv) carbonaceous solid residue is an inert material which does not affect subsequent metallurgical refining operations; (v) the vast majority of brominated phenolic and substituted phenolic compounds are concentrated in the pyrolysis oil.

Nowadays, bitumen is known as a waterproofing material characterized by being a virtually involatile adhesive. It can be derived from crude oil, or present in natural asphalt [9]. Bitumens' characteristics mostly depend on the process of its production and processing, but to the same extent, they are linked to the characteristics of used crude oil.

Before the application of bitumen, it is very important to know its characteristics from various aspects, because of the possible difficulties such as phase discontinuity, instability, or low homogeneity. From the application standpoint, bitumen is a low-cost thermoplastic material widely used in roofing and pavement applications, for making the majority of the paving mixtures, and industrial products that are used over a long time period [10].

This research aims to study the stabilization and possible use of a WPCB vacuum pyrolysis distillate as the filler in the bituminous material to obtain a waterproofing material with acceptable physical and mechanical properties. The use of different easily available and low-cost modifying reagents provided a process for stabilization of the pyrolysis distillate. These reagents are processable and easily incorporated into a bituminous matrix at 2.5, 5, and 10 wt.%. Most of the determined physicochemical and mechanical characteristics are in the range defined by the current standard.

## Experiment

### *Materials*

For this study, the following materials were used: pyrolysis oil obtained in the vacuum pyrolysis, calcium oxide (CaO) bought from limekiln Tara Dobanovci, sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) from Zorka Pharma, sodium hydroxide (NaOH) from NRK engineering, hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) – Zorka Pharma, waste printed circuit boards (WPCBs) from mobile phones and motherboards, Maleic anhydride (C<sub>4</sub>H<sub>2</sub>O<sub>3</sub>) – Acros, *p*-toluenesulfonic acid (C<sub>7</sub>H<sub>8</sub>O<sub>3</sub>S) – Fluka. Bitumen was provided by Interhem Company, Serbia, and data on its properties are given in Table 2 (bitumen from Pančevo refinery).

### *Production of pyrolysis oil*

Collected WPCBs were firstly subjected to the manual removal of batteries and large capacitors. WPCBs prepared in this way were placed in a stainless steel pyrolysis reactor, equipped with temperature and pressure regulators. When the temperature reaches 100 °C, the vacuum pump is switched on. A vacuum of 0.1 ± 0.02 bar is kept constant throughout the process. The vacuum distillation process takes place at a temperature of 400 ± 10 °C for 1 h. As products, the gaseous phase, solid residue, and pyrolysis oil are obtained. This poses a threat to the environment and thus this work is devoted to finding appropriate chemical treatment to transform it into a non-hazardous material possibly used as filler in bituminous products.

### *Pyrolysis distillate treatment with sodium hydroxide (Solidified material 1)*

Into a three three-necked round-bottomed flask (500 ml) equipped with a condenser and a thermometer, 200 ml of pyrolysis distillate was added. In three different tests, 100 ml of 30%, 40%, and 50% solution of NaOH were added with vigorous stirring and heating (200 °C) for 120 min. After cooling, the obtained products solidified. It was found that the addition of 50% aqueous solution NaOH gave the most consistent product without odor.

### *Pyrolysis distillate treatment with calcium oxide (Solidified material 2)*

In three three-necked round bottom flasks, equipped with a mechanical mixer and dropping funnel, 200 ml of waste distillate for three different assays were added. After 30 min and heating at 250 °C with mixing, 25, 35, and 45 g of CaO were added, respectively.

After 135 min, the resulting products solidified. It was found that with the addition of 45 g of CaO a consistent solid product without odor was obtained.

#### *Pyrolysis distillate treatment with sulfuric acid and calcium oxide (Solidified material 3)*

Into three different three-necked, round-bottomed flasks (500 ml) equipped with a condenser, and a thermometer 200 ml of pyrolysis distillate was added. In each of them, 100 ml of 50%, 60%, or 50 ml of concentrated H<sub>2</sub>SO<sub>4</sub> were added, with gentle stirring and heating (250 °C) for 90 min. Thereafter, 30, 45, and 60 g of CaO were added to the flasks, respectively, with constant mixing and heating up at 150 °C for 2 h. The resulting mixtures were left at room temperature overnight to provide material solidification. The material is treated with concentrated H<sub>2</sub>SO<sub>4</sub> and 45 g of CaO showed the best properties.

#### *Synthesis of phenol and malonic acid diesters from calcium salts of oxidative products of oil fraction (Solidified material 4)*

Pyrolysis oil treatment and synthesis of phenol and malonic acid diester were performed in two steps. Extraction of the phenolic component was performed in the following manner: 250 ml of pyrolysis oil was dissolved in toluene (300 ml) and washed two times with 10% NaOH (200 ml). The water extract was acidified to pH=3.5, and the phenolic components were extracted with ether (2 x 200 ml), dried with sodium sulphate, while the ether was distilled to give phenolic component of pyrolysis oil. Maleic anhydride and the phenolic fraction (molar ratio of maleic anhydride and phenol 1:2) were added to a three-necked round-bottomed flask equipped with a thermometer and condenser. After that, 75 ml of xylene was added to the mixture, and 8.1 g of p-TSA (3% by weight) as an esterification catalyst. The reaction was carried out at 150 °C with stirring for 8 h, and the xylene was removed by distillation and used in the following syntheses (solvent recycling). The hot product was poured into the water with vigorous stirring to separate the phenol diester precipitate which was filtered and dried. The aqueous layer is neutralized with CaO, and the resulting precipitate was filtered, dried and combined with the phenol diester product.

The oily fraction (~27 ml) was treated with 10 ml of 30% H<sub>2</sub>O<sub>2</sub> at 50 °C with careful water removal, and afterward into obtained viscous material was added 10 g CaO, followed by heating at 150 °C with stirring for 3 h to give an odorless powdery material. The acid value of the oxidized oily fraction was 320-330 mg KOH/g. Two fractions were combined, mixed and used in the production of bituminous material.

#### *Material characterization*

Fourier transforms infrared (FTIR) spectra were recorded in absorbance mode with a Nicolet™ iS™ 10 FT-IR Spectrometer (ThermoFisher SCIENTIFIC) equipped with Smart iTR™ Attenuated Total Reflectance (ATR) Sampling accessories.

Physicochemical characteristics of bituminous materials were determined by using the standard methods given in Table 2.

For the determination of the ion mobility the Toxicity characteristic leaching procedure – TCLP (Method 1311) was applied. The procedure for TCLP test includes two types of extraction media:

1. Extraction medium 1: 5.7 ml of glacial acetic acid was added to 500 ml of distilled water, and 64.3 ml of 1 M NaOH and diluted to 1000 ml (pH value  $4.93 \pm 0.05$ );

2. Extraction medium 2: 5.7 ml of glacial acetic acid was diluted with distilled water to a volume of 1000 ml (pH of the solution  $2.88 \pm 0.05$ ).

Water content determination in mas.% (EPA 9000:2007), metal content in mg kg<sup>-1</sup> (EPA 3051A/EPA 6010c:2007), polycyclic aromatic compound in mg kg<sup>-1</sup> (EPA 3550C/8270D:2007), polychlorinated biphenil content in mg kg<sup>-1</sup> (EN 12766-1:2000), halogen element and sulfur in mg kg<sup>-1</sup> (DML 5.6:2014) and GC-MS analysis was performed in Anahem Laboratory.

## Results

Based on the results of the analysis of pyrolysis oil, the contents of major constituents are given in Table 1. Other parameters of pyrolysis distillate: water content was <3%, flash point 46 °C, heavy metals and metalloids were at the level lower than prescribed according to the Rulebook on categories, testing and classification of waste, Official Gazette of RS, 56/2010 and 93/2019.

Table 1. Composition of the main component of oil resulting from the pyrolysis of WPCBs.

Component	content, % *
Benzofuran	1.24
2-methyl-2,3-dihydrobenzofuran	2.38
2-methyl-benzofuran	1.56
2,3-dimethyl-benzofuran	1.12
<i>o</i> -bromophenol	4.96
Phenol	35.68
<i>o</i> -ethylphenol	2.36
<i>p</i> -methylphenol	6.22
2-(1-methylethyl) phenol	2.04
<i>m</i> -ethylphenol	6.67
4-(1-methylethyl) phenol	16.47
2,6-dibromophenol	0.44
1-hydroxy-2,4-dibromobenzene	2.05
<i>p</i> -ethylphenol	1.88
<i>m</i> -isopropylphenol	4.66
Aliphatic fraction	10.27

\* All data was recalculated from mg kg<sup>-1</sup> to mass%; All values of metals and metalloids concentration are lower than prescribed by EPA 3051A/EPA 6010c:2007, except 634 mg kg<sup>-1</sup> for Sn (reference value 100 mg kg<sup>-1</sup>)

The results in Table 1 show that the composition of phenols contained in the pyrolysis oil are in accordance with literature data [11, 12, 13]. Due to the high phenol content, different modifications of pyrolysis oil were performed in order to achieve stabilized products that can be used as fillers for hydro-insulating materials based on bitumen. In the first step, FTIR spectra of obtained solidified materials were recorded and given in Figure 1.

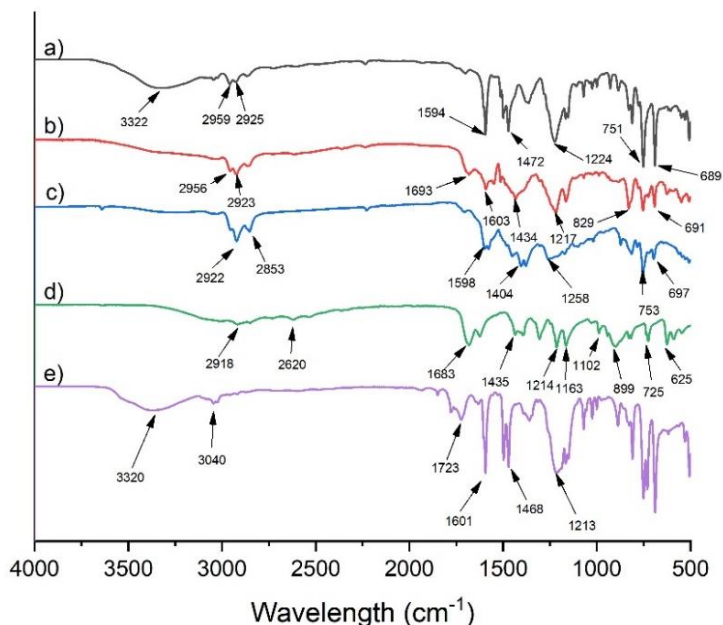


Fig. 1. FTIR spectrum of: a) the WPCB distillate; b) Solidified material 1; c) Solidified material 2; d) Solidified material 3; and e) Solidified material 4.

### Discussion

The FT-IR spectrum given in Figure 1a) clearly shows bands at  $2958\text{ cm}^{-1}$  corresponding to asymmetric stretching vibrations of the methyl group, and a band at  $2923\text{ cm}^{-1}$  corresponding to asymmetric stretching vibrations of the methylene group. Weak bands observed at values  $> 3000\text{ cm}^{-1}$  correspond to stretching vibrations of the  $=\text{CH}$  group present in phenols. Deformation vibrations (scissoring) of the methylene group, overlapped with asymmetric deformation vibrations of the methyl group, was observed at  $1472\text{ cm}^{-1}$ . Also, deformation vibrations of the methylene group, wagging and twisting, and symmetric deformation vibrations of the methyl group was observed at  $1071\text{ cm}^{-1}$  and  $1376\text{ cm}^{-1}$ , respectively. The bands at  $751$  and  $689\text{ cm}^{-1}$  originate from out-of-plane deformation vibrations from aliphatic and aromatic structures.

Figure 1b) shows the FTIR spectrum of Solidified material 2. The bands observed at  $1693$  and  $1603\text{ cm}^{-1}$  are characteristic for skeletal  $\text{C}=\text{C}$  aromatic vibrations. The wide intense bands at  $1434$  and  $1217\text{ cm}^{-1}$  originate from the C-O vibration present in the phenoxide structure. The bands in the range  $829$  to  $691\text{ cm}^{-1}$  originate from deformation vibrations out-of-plane of aromatic and aliphatic structures.

The FTIR spectrum is given in Figure 1c) shows changes in the structure of the distillate when it is treated with calcium oxide. The wide intense bands at  $1404$  and  $1258\text{ cm}^{-1}$  originate from the C-O vibration present in calcium phenoxide.

Figure 1 d) shows wide intense bands at  $1163$  and  $1006\text{ cm}^{-1}$  originating from the calcium salt phenol and aliphatic sulfonic acids. The absence of O-H stretching vibrations indicates successful neutralization with CaO for both solidified materials 2 and 3.

Figure 1e) shows the changes in structure at  $3320\text{ cm}^{-1}$  a peak characteristic for OH stretching vibrations present in carboxylic acids, at  $3040\text{ cm}^{-1}$  is the peak for the CH stretching vibrations present in an unsaturated structure. Also, at  $1776$  and  $1723\text{ cm}^{-1}$  stretching vibrations of carbonyl bond present in phenyl esters. The C–O stretching vibrations present in esters is shown at the wavelength of  $1213\text{ cm}^{-1}$ .

In the next step, the obtained products were incorporated into bitumen. The mixing process consisted of compounding softened bitumen (at  $100^\circ\text{C}$ ) using a mechanical stirrer and adding of 2.5, 5, 7.5, and 10 wt.% by weight of the ground mass from the experiment in which the distillation oil was treated with concentrated sulfuric acid, for 15 min. Mechanical tests of bituminous materials were performed, and the obtained characteristics are given in the following Tables 2-5.

Table 2. Physicochemical and mechanical properties of bitumen.

Characteristic	Unit	Result	Range	Method
Specific density at $25^\circ\text{C}$	$\text{kg/m}^3$	1018		ASTM D 70-76
Hardness at $25^\circ\text{C}$ , 5 s	0.1 mm	67	60-79	
Softening point	$^\circ\text{C}$	50	48-58	
First drop pint	$^\circ\text{C}$	318		ASTM D 92-02
Flash point	$^\circ\text{C}$	313	min. 232	
Rigidity $25^\circ\text{C}$ , 5 cm/min	Cm	138	min. 100	ASTM D 113-99
Density	$\text{kg/m}^3$	1022	min. 1	
Paraffin concentration	%	0.6	max. 20	
Solubility in DCM	%	99.1	min. 99	
Penetration at $25^\circ\text{C}$ , 1/10 mm	Mm	64.1	60-70	ASTM D 5-97
Viscosity at $135^\circ\text{C}$	Pa s	0.332		ASTM D 4402
Mass loss during heating	%	0.0570		ASTM D 6-80

Table 3. Physicochemical and mechanical properties of bituminous material with 5 and 10 wt. % of Solidified material 2 (Experiment 2.4).

Characteristic	Unit	5 wt.%	10 wt.%	Range	Method
Specific density at $25^\circ\text{C}$	$\text{kg/m}^3$	1018,5	1019		ASTM D 70-76
Hardness at $25^\circ\text{C}$	mm	67,3	68,5	60-79	
Softening point	$^\circ\text{C}$	50,5	50,9	48-58	
First drop pint	$^\circ\text{C}$	313,7	314,9		ASTM D 92-02
Flash point	$^\circ\text{C}$	315	319	min. 232	
Rigidity at $25^\circ\text{C}$ , 5 cm/min	Cm	128	125	min. 100	ASTM D 113-99
Density	$\text{kg/m}^3$	1023	1024	min. 1	
Paraffin concentration	%	2,7	5,1	max. 20	
Solubility in DCM	%	97,9	89,6	min. 99	
Penetration at $25^\circ\text{C}$ , 1/10 mm	Mm	63,7	62,4	60-70	ASTM D 5-97
Viscosity at $135^\circ\text{C}$	Pa s	0,334	0,338		ASTM D 4402
Mass loss during heating	%	0,0568	0,0567		ASTM D 6-80

Similar values of all determined parameters (differences in the range  $\pm 2.2\%$ ) were found for bituminous material with Solidified material 3 due to similar product properties obtained by CaO and H<sub>2</sub>SO<sub>4</sub>/CaO treatment (Experiment 2.4 and 2.5). According to solubility in DCM it does not have market value as a hydro insulating material, but it could be used in the production of asphalt mixture for road building. Bituminous material obtained by incorporation of Solidified material 1 also showed a similar change of the physicochemical and mechanical properties to one given in Tables 3 and 4, but the results from TCLP test showed that some parameter exceeds permitted limits (Table 6).

Table 4. Physicochemical and mechanical properties of bituminous material with 5 and 10 wt. % of Solidified material 4 (Experiment 2.6).

Characteristic	Unit	Result for 5%	Result for 10%	Limit range	Method
Specific density at 25 °C	kg/m <sup>3</sup>	1020	1009		ASTM D 70-76
Hardness at 25 °C, 5 s	0.1 mm	73,9	61,8	60-79	
Softening point	°C	58,5	52	48-58	
First drop pint	°C	317,2	302		ASTM D 92-02
Flash point	°C	342	386	min. 232	
Rigidity 25 °C, 5 cm/min	Cm	116	133	min. 100	ASTM D 113-99
Density	kg/m <sup>3</sup>	1090	1019	min. 1	
Paraffin concentration	%	0,7	1,2	max. 20	
Solubility in DCM	%	99,1	99,0	min. 99	
Penetration at 25 °C, 1/10 mm	Mm	60,5	67,9	60-70	ASTM D 5-97
Viscosity at 135 °C	Pa s	0,308	0,328		ASTM D 4402
Mass loss during heating	%	0,0424	0,0553		ASTM D 6-80

Based on the results shown in Tables 2 - 4, it can be seen that the materials produced by mixing of the solidified material 2 with bitumen show better mechanical properties than non-modified bitumen. With the increase of the content of the filler (from 2.5 to 10 %), the mechanical properties of the waterproofing materials are improved. On the other hand, when the solidified material 4 was used as a filler for bitumen, the acquired material showed good characteristics at 10% mass content.

Hazardous characteristic of the obtained bituminous materials was determined according to TCLP test. The obtained results are given in Table 5.



Table 5. Concentrations of leachate in Extraction medium 2 after TCLP test ( $\text{mg L}^{-1}$ ).

Experiment	As	Sn	Cd	Cr	Cu	Ni	Pb	Sb	V	Zn
2.3	17	840	4	17	55	2.5	20	52	4	300
2.4	16	800	2	16	50	20	24	48	60	500
2.5	6	400	0,5	8	20	4	15	41	41	420
2.6	2	100	0,5	8	10	11	10	21	18	280
Limit	20	1000	5	300	100	500	100	50	200	1000

\*Ordinance on categories, testing and classification of waste ("Official Gazette of RS", No. 56/2010, 93/2019 and 39/2021); result of TCLP showed increased concentration of phenol  $>550 \text{ mg L}^{-1}$  (determined using UV-Vis method; recalculated to phenol)

According to the results from Table 5, it can be concluded that the content of all tested samples from experiments 2.4 - 2.6 have a concentration of metals below the limits prescribed by law, and as such, do not pose a danger to the environment. On the contrary, the results obtained by examining the samples from experiment 2.3 show that the estimated concentrations of phenol are  $>550 \text{ mg L}^{-1}$ , and thus Solidified material 1 (Experiment 2.3) could not be considered for further use in practice.

## Conclusion

This paper presented the results of the use of solidified pyrolysis distillate from WPCBs for the production of waterproofing bituminous materials. The composition of the WPCB distillate was determined using GC-MS analysis, while FTIR spectroscopy was used for the characterization of solidified pyrolysis distillate. The solidified material was used as a filler at 2.5, 5, 7.5, and 10 wt.% addition into bitumen. The determination of physicochemical and mechanical properties of obtained bituminous materials, as well as the results from TCLP test, proved their possible use as waterproofing materials which satisfy both current regulations and environmental protection. Due to the acceptable results from TCLP test, except for the phenols leaching from the bituminous material with Solidified material 1, the negative environmental impact of the pyrolysis distillate was eliminated by applied treatments. The bituminous material with the best characteristics was obtained by the addition of Solidified material 4 at 10 wt.%, which, in that way, defines the future direction of the development of the pyrolysis distillate treatment technology. The presented study is a part of the modern trend related to polemics on energetic or other methods of the exploitation of waste material, i.e. plastic waste and WPCBs, in order to attain economic benefit, i.e. market products, with the satisfaction of current legislative. It is crucial that the proposed technology is a good achievement related to dangerous waste treatment producing non-hazardous stabilized materials applicable in bituminous material production. The developed methodology satisfies the principles of waste management, material recycling, resource preservation, and environmental protection. Future development will relate to the production of asphalt mixtures ready to use and tested in a laboratory and on the site of application.

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