

## COPOLYMERIZATION OF INDUSTRIAL ORGANOCHLORINE WASTE WITH WOOD PYROLYSIS PRODUCTS FOR BITUMEN COMPOSITIONS

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<https://doi.org/10.23939/chcht17.04.929>

**Abstract.** The work presents a new approach to the copolymerization and co-processing of industrial organochlorine waste (OCW) resulted in the production of vinyl chloride at Karpatnaftochim LLC, Kalush, Ukraine, and liquid wood pyrolysis products (LWPP) generated by the activated charcoal production at Perechyn Forestry and Chemical Plant, LLC. Kalush, Ukraine. This article highlights studies of the composition of the raw materials and the properties of the resulting products. Through the use of infrared spectroscopy and DTA analysis, the primary structure and properties of copolymers, oligomers, asphalt and bitumen products obtained from OCW and LWPP were determined. It describes the trial of the liquid products of wood pyrolysis in bitumen compositions with OCW.

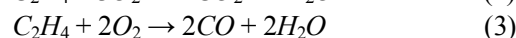
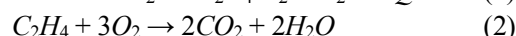
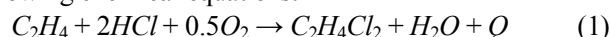
**Keywords:** organochlorine, waste, product, pyrolysis wood, vinyl chloride, 1,2-dichloroethane, bitumen, asphalt, compositions, formaldehyde, composites.

### 1. Introduction

Over the past 10-15 years, the industry has expanded its sales markets of polyethylene (PE) and polyvinyl chloride (PVC) polymers up to 15 million tons. These polymers have replaced metal and wood products used for packaging, pipes, various coatings, furniture, profile windows, doors, and other structures. The increase of the production capacity of organochlorine monomers and polymers causes the increase of the amount of industrial organochlorine waste (OCW). The production of vinyl chloride and its derivatives generates up to 1.5 million

tons of OCW per year, and this index is growing worldwide. The authors of the article proposed a new method of integrated processing and utilization of OCWs by copolymerizing them with a heavy fraction of liquid wood pyrolysis products (LWPP (production name "pulp")). The purpose of our work is to improve the existing methods and develop new ways of processing and utilization of liquid industrial organic (LWPP) at the Perechyn Forestry and Chemical Plant with organochlorine waste (OCW) of Karpatnaftochim LLC, Kalush, Ukraine. The results of the study can be used for production of cheap and non-toxic polymer that can be applied as a component of waterproofing, asphalt, and anti-corrosion coatings in bitumen compositions.

The industrial process of oxidative chlorination of ethylene (OCE) is the most efficient in the industrial production of 1,2-dichloroethane (1,2-DCE) and vinyl chloride monomer (VCM) at Karpatnaftochim LLC in Kalush, as it allows to achieve 99% conversion of raw materials and 98.8% selectivity in terms of 1,2-dichloroethane yield.<sup>1</sup> At the same time, in addition to 1,2-DCE, a number of organochlorine wastes are formed.<sup>2</sup> The reactions of ethylene oxidation to 1,2-DCE ( $C_2H_4Cl_2$ ) and ethylene oxidation (combustion) proceed according to the following chemical equations:<sup>2</sup>

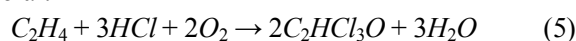


In this case, side reactions can occur, which form the following OCWs products:

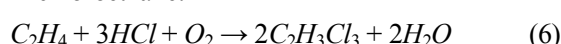
1. Carbon tetrachloride:



2. Chloral:



3. 1,1,2-Trichloroethane:



and other impurities – 1-2% of the obtained DCE.

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## 2. Experimental

The approximate chemical composition of OCWs (gaseous, liquid and solid organochlorine wastes) at the Karpatnaftochim production facility in Kalush, Ukraine, determined by chromatographic method, is shown in Table 1. OCWs contain more than > 30% of the total a residue of unidentified polychlorinated OCWs. These OCWs also contain small amounts of hydrogen chloride and chlorine (in the range of millionth of a percent). The amount of moisture in the OCWs does not exceed 20-200 ppm.<sup>3</sup>

**Table 1.** Procedure for the yield of CWA components in a chromatographic analysis

№	Component	Time, min
1	ethylene	0.95
2	vinyl chloride	1.4
3	ethyl chloride	1.8
4	trans-1,2-dichloroethylene	3.45
5	carbon tetrachloride + 1,1-dichloroethane	4.0
6	methylene chloride	4.65
7	benzene	5.15
8	1,1,2-trichloroethylene + cis-1,2-dichloroethylene	6.35
9	chloroform	7.05
10	perchloroethylene	8.2
11	1,2-dichloroethane	10.0
12	chlorobenzene	15.45
13	1,1,2-trichloroethane + 1,1,1,2-tetrachloropropane	16.5
14	1,2,3-trichloropropane	26.75
15	1,1,2,2-tetrachloroethane	29.6
16	1,2-dichlorobenzene	31.2
17	chlorex	34.0
18	Σ HOV	>35.0

### 2.1. Methods of analysis

Gas-liquid chromatography was the main means of analyzing OCW compounds. The analysis of OCWs in the study of OCW chlorination was performed on a chromatograph of the "Tsvet" type with a flame ionization detector under the following conditions of analysis: (1) length of the working column 3 m; column diameter 3 mm; sorbent – 12% PEG 1500 on 3 K color chromium; working column temperature – 373 K and 423 K, set 1.25 min after the peak of 1,2-DCE; evaporator temperature – 463 K; analysis time – 50 min; helium was used as a car-

rier gas; chromatograms were calculated by the method of internal normalization.

The order and time of the components on the OCW chromatogram are shown in Table 1. The change in the composition of OCW during the study of alkaline dehydrochlorination and polycondensation of OCW with polysulfides (PSF) of alkali metals was analyzed according to TUU-24.1.-31256759-005-2004, on a gas-liquid chromatograph Carlo Erba GC 8000 series with a flame ionization detector (steel column d = 3 mm, l = 3 m, filled with a phase of 25% emulsogen on the chromosorb). The temperature of the thermostat is in the range from 55 to 413 K. Detector temperature is 473 K, injector (evaporator) temperature = 423 K. Carrier gas consumption: helium - 135 KPa, air - 130 KPa, hydrogen - 75 KPa. The sample volume was 0.8 μL. Peak areas were calculated automatically.<sup>4</sup>

### 2.2. Determination of polymer resin curing time with OCW and LWPP on the Vick device

The OGTs-1 device is designed to determine the normal density and setting time of cement dough (including asphalt) in accordance with GOST 310.3-76. For the test, a free movement of the dough and the permissible zero deviation are checked first. The test surface is cleaned and checked for curvature. The needle of the device is immersed in the test sample of the product of a given density several times, clensing the needle after each immersion.

Oxidized bitumen of the BND 70/100 brand according to DSTU 4044:2019, selected at PJSC "UKRTATNAFTA" (Kremenchuk, Ukraine), was used for the study.<sup>5</sup> Bitumen-polymer mixtures were prepared using a Daihan Scientific HT-50 DX mixer (Daihan Scientific Co., Ltd, Korea) equipped with an impeller in the form of a six-blade turbine. A sample of the initial bitumen (650 g) was placed in a metal container and heated with stirring on an electric stove to the required temperature. After reaching the temperature with constant stirring of the sample, the modifier was gradually added in the required amount.

## 3. Results and Discussion

### 3.1. General characteristics of liquid wood pyrolysis products (LWPP)

Of all the thermal methods of wood processing, high-temperature wood pyrolysis is the most important. Wood pyrolysis involves the decomposition of wood

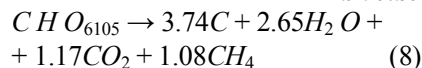
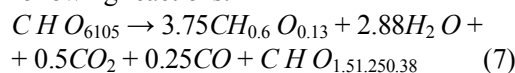
through heating to 723 K in the absence of air, resulting in the production of gaseous and liquid products, including wood resin, along with a solid residue known as charcoal. Wood pyrolysis is typically conducted without air access at temperatures up to 723 K in large-scale facilities, such as the Perechyn Forest and Chemical Plant. This plant primarily produces charcoal, acetic acid, and their derivatives, also generating liquid wood pyrolysis products (pulp) as a byproduct. Solid products of wood pyrolysis consist of cellulose residues,  $(C_6H_{10}O_5)_x$  natural polymer, and oxygen-containing hydrocarbons-polysaccharides; they also contain a small amount of nitrogen-containing substances.<sup>6</sup> When birch wood is pyrolyzed to a moisture content of 8-10%, the yield of solid wood is 280-295 kg/m<sup>3</sup> of wood, and 350-380 kg/m<sup>3</sup> of solid wood is obtained from air-dry wood. The volume of well-cooked charcoal decreases and is approximately 60% of the volume of the initial wood fed for pyrolysis.<sup>7</sup> To extract acetic acid, LWPP is usually de-alcoholized and desaturated. Depending on the method used, the following useful products can be extracted from 350-380 kg of "pulp", in kg: 1) Polymer resin 22-29 kg; 2) Acetic acid 18-22 kg; 3) Alcohol-containing products 7-10 kg. Based on 1 m<sup>3</sup> of pyrolyzed wood, the recovery rate for all acids can be 66-75% and for the sum of alcohol products - 60-70%. But mostly at most enterprises, LWPP is burned in the same furnaces that heat wood and produce charcoal.

The liquid products of wood pyrolysis were obtained from Perechyn Chemical Plant, which serves as a supplier to the largest activated carbon plants in Europe. Perechyn Chemical Plant products are regularly stocked by renowned European manufacturers such as LIDL, Kaufland, Carrefour, EDEKA, and TESCO. The plant holds impressive certifications that would be envied by many industrial enterprises in Ukraine, operating in accordance with BSCI, PEFC™, and ISO standards. The characteristics of the produced charcoal include a carbon content of up to 93% C, humidity ranging from 3% to 8% H<sub>2</sub>O, ash content ranging from 2% to 15%, and a particle size fraction of 0-80 mm.<sup>7</sup>

### 3.2. The process of wood pyrolysis with separation into fractions and chromatographic composition of LWPP

Wood pyrolysis is conducted by heating wood to a temperature of 723 K, leading to its decomposition. The process of wood pyrolysis relies on free radical reactions that involve the thermal breakdown of hemicellulose, cellulose, and lignin. These reactions occur within specific temperature ranges: hemicellulose at 473-533 K, cellulose at 513-623 K, and lignin at 523-673 K. At a temperature

of 593 K, the ratio of rate constants for these reactions is approximately 10:1:0.25. The composition of charcoal obtained from wood pyrolysis typically includes 80-92% of carbon (C), 4.0-4.8% of hydrogen (H), and 5-15% of oxygen (O). The pyrolysis process can be described by the following reactions.<sup>8</sup>



The pyrolysis of wood is an exothermic process that releases a large amount of heat (1150 kJ/kg). The raw material chosen for the synthesis was LWPP from Perechyn Forestry and Chemical Plant LLC, the largest Ukrainian and leading European producer of charcoal products with more than a century of production history. At the end of 2019, the monthly production volume was up to 4000 tons of charcoal. The raw material is 100% hardwood from Ukrainian forests. As a result of wood pyrolysis, the following 3 fractions are formed: 1) density at 293 K 1,23 kg/dm<sup>3</sup>; 2) pH 4.5; 3) Brookfield viscosity at 293 K (LV-3, 60 RPM): 377 mPa-s; 4) Water content 6.2%.<sup>9</sup> Determination of heavy resin parameters was carried out for density according to the method of DSTU ISO 2811-1:2019, pH indicator of hydrogen ion activity according to DSTU EN 12850:2020, dynamic viscosity was determined according to DSTU EN 13302:2019, determination of water content according to DSTU ISO 3733:2017, Table. 2.

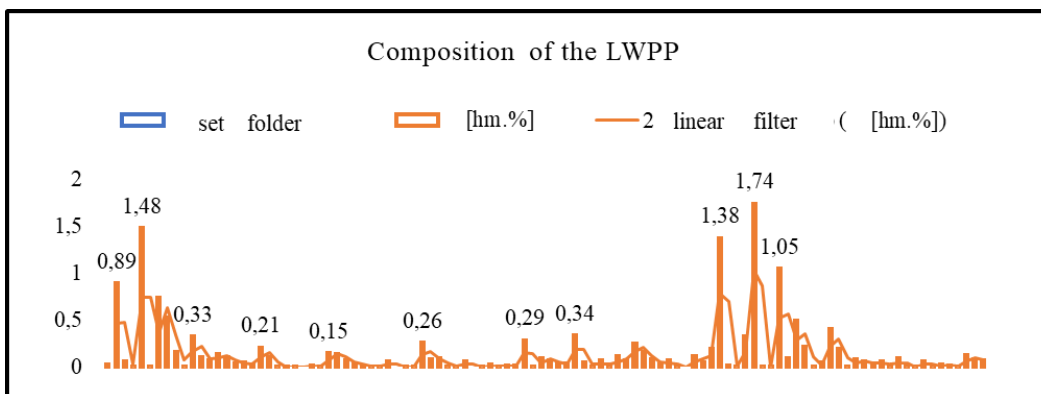
Fraction №2 (383-393 K), resin from wood pyrolysis to charcoal of GOST 7657-84, obtained after vacuum distillation of water and other substances at 393 K from heavy wood pyrolysis resin has the composition 1) Boiling point  $\geq 393$  K; 2) Density at 293 K, 1.320 kg/dm<sup>3</sup>; 3) Hydrogen index, pH 5.1; 4) Brookfield viscosity at 293 K (LV-4, 20 rpm): 1314 mPa-s.

Fraction №3. The hydrophobic part had a light yellow color, which eventually changed to red-brown, indicating that the reaction was underway. Thus, these may be light and oxidation-sensitive aromatic hydrocarbons.

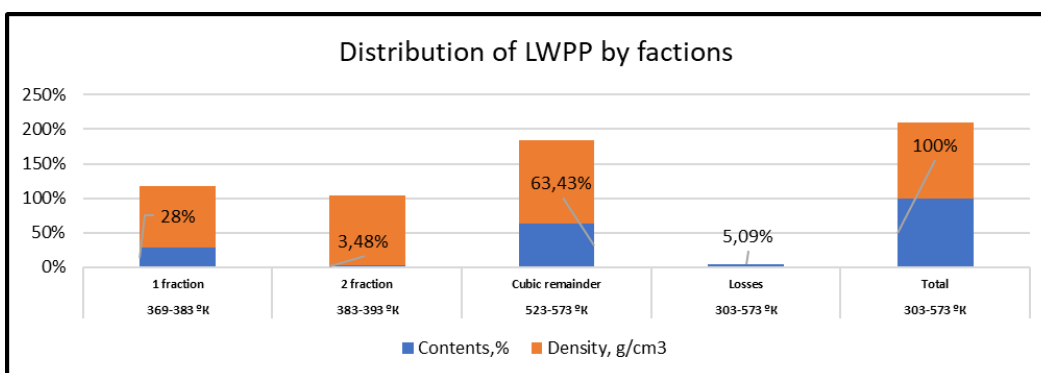
Water and light fractions were extracted through a vacuum process. A vacuum compressor was utilized to reduce the pressure to 30 kPa, while a water bath maintained a temperature of 341-344 K. The following results were obtained for the composition of fraction No. 3: 1) 4.78-5.1% water and water-soluble compounds of the distillation; 2) 1.75-2.2% hydrophobic part of the distillation - a mixture of benzene, phenols and their homologues; 3) 89.1-90.6% residue of dehydrated organic compounds; 4) distillation losses of 2.2-2.5%.

**Table 2.** Chromatographic composition of LWPP, Perechyn Timber and Chemical Plant

No. p/n	HARDWOOD	Oak, hornbeam, birch.	Dimensionality
1		<b>Water Content</b>	<b>4.1</b>
		<b>dry: wt. % based on oil weight without water content (</b>	<b>Anhydrous Mass</b>
	CAS-No.	<u><b>Compound</b></u>	<u><b>wt.% wet</b></u>
1.		<b>Acids</b>	<b>4.182</b>
2.	64-19-7	Acetic acid	4.182
3.		<b>Nonaromatic Esters</b>	<b>1.409</b>
4.		<b>Nonaromatic Ketones</b>	<b>1.842</b>
5.		<b>Furans</b>	<b>1.045</b>
6.		<b>Benzenes</b>	<b>11.721</b>
7.	108-88-3	Toluene	0.157
8.		<b>Aromatic Ketones</b>	<b>0.106</b>
9.	98-86-2	Acetophenone	0.106
10.		<b>Lignin derived Phenols</b>	<b>8.505</b>
11.	108-95-2	Phenol	1.472
12.	95-48-7	Cresol, o-	1.517
13.		<b>Guaiacols (Methoxy phenols)</b>	<b>2.888</b>
14.	5932-68-3	Guaiacol, 4-propenyl-(trans) (Isoeugenol)	1.603
15.		<b>Syringols (Dimethoxy phenols)</b>	<b>5.906</b>



**Fig. 1.** Chromatogram of a sample of liquid wood pyrolysis products (LWPP) from Perechyn Forestry and Chemical Plant



**Fig. 2.** Fractional distribution of LWPP according to a boiling point

**Table 3.** Composition of the copolymer resin OCW and LWPP

Component	V, mL	% wt.
OCW	10	47.62
LWPP	10	47.62
Catalyst and or initiator	1	4.76

**Table 4.** Physical and mechanical properties of oxidized bitumen with a copolymer of OCW and LWPP

Indicator	BND 70/100	Bitumen + 3 % by weight. Sopolim. OCW and LWPP	Bitumen + 6 % wt. of a copolymer of OCW and LWPP
Softening point, K	320.35	321.35	320.55
Penetration at 298 K, 0.1 mm	82	85	75
Ductility at 298 K, cm	>150	>150	117
Adhesion to the glass surface, %.	29	60	77
Adhesion to the surface of crushed stone, %.	3.0	4.0	4.5

### 3.3. Copolymerization of OCW with the LWPP fraction

For the copolymerization of organochlorine waste (OCW) with the LWPP fraction, a dedicated laboratory setup was utilized, employing a 1-liter autoclave reactor constructed from stainless steel. The schematic diagram of this installation can be found in the referenced literature.<sup>4</sup> Using the data acquired, the optimal parameters for synthesizing a copolymer of OCWs and LWPPs were determined.

### 3.4. Results of the catalytic copolymerization of LWPP and OCW

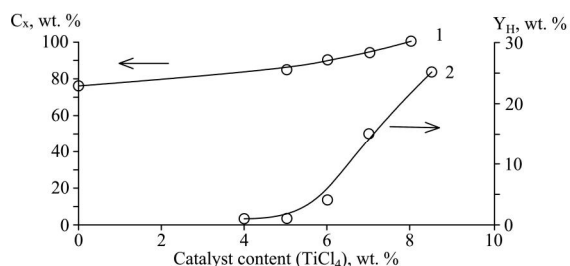
The polymerization was conducted using a 1:1 ratio of the components of organochlorine waste (OCW) and the dehydrated fraction of liquid wood pyrolysis products (LWPP). The polymerization reaction duration was 2 hours. After that, a resinous product is formed. As a result of studying almost all the operating parameters, the following optimal conditions for the process were established, which are summarized in Table 3. The volume ratio of pyrolysis resin and OCW is 1:1; The content of titanium tetrachloride is 4.5-5% by weight of OCW with LWPP. The temperature of the copolymerization process is  $\geq 393$  K.

Based on the copolymerization of DGC products of OCW and the dehydrated fraction of LWPP, a preliminary recipe for the production of polymer resin was developed. The main components of the resin are outlined in Table 4.

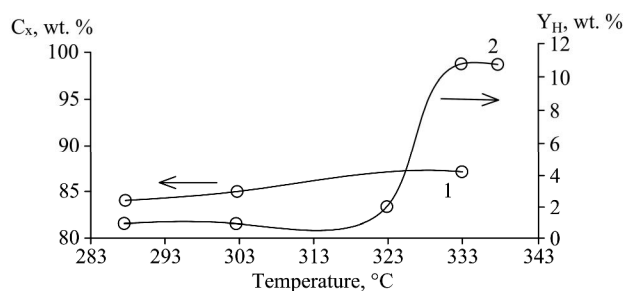
In the presented graphs No. 3 and 4, the volume ratio of OCW produced by VC and the unsaturated fraction of LWPP was maintained at 1:1; the volume ratio of OCW produced by VC and solvent was equal to 3:1; the

solvent was xylene. From the obtained dependence of the conversion of OCW and the LWPP fraction on the concentration of the cationic catalyst (Figure 4, curve 1), one can observe a regularity, with an increase in catalyst concentrations from 1 to 8%, the conversion of monomers increases from 78% to 99%, respectively. When the catalyst content is increased above 8%, the conversion of monomers (unsaturated) does not change. Provided that in the copolymerization reaction, in addition to mutually soluble polymers and oligomeric products, it is possible to form insoluble polymers in xylene and OCW. We studied the dependence of polymer yields on the amount of catalyst and temperature. The results shown in Figures 3 and 4 (curve 2) confirm this. From the results presented, it can be seen that the amount of insoluble chloropolymer does not exceed 20-25%.

In this case, an increase in temperature from 288 K to 333 K has a greater effect on the intensification of the copolymerization process, since its increase gives an increase in the yield of insoluble polymer from 1 to 11% (Fig. 4, curve 2). To further explore this, we tested the effect of temperature factors on the conversion of OCW and the LWPP fraction Cx (curve 1 of Fig. 4) and the yield of insoluble polymer Yn as a function of temperature (curve 2), as shown in Figure 4. An increase in temperature from 298 K to 341 K gives a slight acceleration to the polymerization reaction rate, which is expressed by an increase in conversion from 74% to 87% in this temperature range. This may be due to the ionic mechanism of this copolymerization reaction of the LWPP fraction and OCW. The amount of catalyst has a greater effect. At the same time, an increase in temperature from 323 K to 338 K increases the yield of insoluble polymer compounds by 6 times. The obtained oligomeric resin products were used to produce modified bitumen-asphalt road compositions.



**Fig. 3.** Dependence of the conversion ( $C_x$ ) of OCW waste and LWPP fraction (curve 1) and the yield ( $Y_H$ ) of insoluble polymer (curve 2) on the catalyst concentration ( $TiCl_4$ ) 4 % by weight, at 323 K



**Fig. 4.** Dependence of the conversion ( $C_x$ ) of OCW waste and LWPP fraction (curve 1) and the yield ( $Y_H$ ) of insoluble polymer (curve 2) on the temperature of the copolymerization reaction at  $TiCl_4$

### 3.5. Modification of road petroleum bitumen with copolymer resin OCW and LWPP and products of polycondensation of this resin with formaldehyde

Initially, we studied the copolymer resin of OCW and LWPP as a component of road oil bitumen. The physical and mechanical properties of oxidized bitumen with different amounts of copolymer of OCW and LWPP are presented in Table 4. Based on the data in Table 4, the copolymer of OCW and LWPP can be used as a component of road bitumen, since it does not deteriorate the basic operational properties of bitumen, but at the same time improves adhesion properties (improves adhesion to glass and crushed stone surfaces). That shows that the use of such an additive will eliminate the need to add expensive adhesives to bitumen.<sup>5</sup>

In order to obtain a polymeric bitumen modifier and bind phenols contained in LWPP, polycondensation of a copolymer of OCW and LWPP with formaldehyde was performed. The material balance of the polycondensation of the copolymer of OCW and LWPP with formaldehyde is shown in Table 5<sup>10</sup>. The polycondensation reaction temperature is 373 K, the duration is 1 hour. The physical and mechanical properties of bitumen-polymer mixtures obtained using the synthesized polymer are given in Table 6.

**Table 5.** Material balance of polycondensation of a copolymer of OCW and LWPP with formaldehyde

No. of the polymer composition (P)	№1	№2	№3
Downloaded, g			
Copolymer of OCW and LWPP	30.24	30.17	30.2
Formalin (37% p.o. formaldehyde)	5.3	7.52	10.52
Concentrated HCl	1.45	1.52	1.57
Total	36.99	39.21	42.29
Received, g			
Polymer	<b>18.99</b>	<b>19.99</b>	<b>20.57</b>
Volatile components (vacuum)	18.00	19.22	21.72
Total	36.99	39.21	42.29
The yield of the copolymer of OCW and LWPP, % wt.	62.7	66.2	68.1

**Table 6.** Physical and mechanical properties of bitumen-polymer mixtures

Indicator	BND 70/100	BND 70/100+ 3 % wt. №1	BND 70/100+ 3 % wt. №2	BND 70/100+ 3 % wt. №3
Softening point, K	320.35	321.55	320.95	320.55
Penetration at 298 K, 0.1 mm	82	55	78	83
Ductility at 298 K, cm	>150	89	123	137
Adhesion to glass, %	29	78	72	48
Adhesion to a crushed stone, %	3.0	5.0	4.5	3.5
Homogeneity	homogeneous	homogeneous	homogeneous	heterogeneous

Based on the data presented in the Table 4-6, it is evident that a heterogeneous binder is obtained when using polymer №3, i.e., the synthesis of a polymer modifier should be carried out at a mass ratio of formalin to the copolymer of OCW and LWPP of less than 1/3.

## 4. Conclusions

The paper provides a theoretical justification and solution of the scientific problem, to improve existing technologies, develop new ones, simpler, and more economical technologies for the integrated processing and utilization of industrial organochlorine waste and unsaturated resins from charcoal pyrolysis, namely, the liquid product of wood pyrolysis ("pulp"), which helps to preserve hydrocarbon raw materials with increased environmental safety of technological processes and reduce the amount of waste in industrial processes of producing DCE, VC, EP, and charcoal.

It has been established that liquid wood pyrolysis products (LWPP) can be used together with CWP as a component of bitumen compositions, as well as for polycondensation reactions with formaldehyde to introduce this modifier into bitumen in an amount of 1-6%, which improves the physical and mechanical properties and reduces the cost of bitumen compositions.

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Received: July 03, 2023 / Revised: August 27, 2023 / Accepted: September 14, 2023

## КОПОЛІМЕРИЗАЦІЯ ПРОМИСЛОВИХ ХЛОРОРГАНІЧНИХ ВІДХОДІВ З ПРОДУКТАМИ ПІРОЛІЗУ ДЕРЕВИНИ ДЛЯ БІТУМНИХ КОМПОЗИЦІЙ

**Анотація.** У представленій роботі запропоновано новий підхід до проведення кополімеризації та сумісної переробки промислових хлорорганічних відходів (ХОВ) виробництва вінілхлориду ТОВ «Карпатнафтохім» м. Калуш, Україна, та рідких продуктів піролізу деревини (РППД), які утворюються при виробництві активованого вугілля з деревини на ТДВ «Перечинський лісохімічний комбінат». Вивчено склад сировини та властивості отриманих продуктів. ІЧ-спектроскопією та ДТА аналізом встановлено первинну будову та властивості одержаних із ХОВ і РППД кополімерів, олігомерів та асфальтових і бітумних виробів з них. Проведено випробування рідких продуктів піролізу деревини в складі бітумних композицій разом з ХОВ.

**Ключові слова:** хлорорганічні, відходи, продукт, піроліз деревини, вінілхлорид, 1,2-дихлоретан, бітум, асфальт, композиції, формальдегід, композити.

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