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RECOVERY OF PHENOL-FORMALDEHYDE SOLID WASTE FROM THE PRODUCTION OF INORGANIC THERMAL INSULATING MATERIALS

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Abstract. *The production of inorganic thermal insulation materials with a fibrous structure, consisting of a fibrous skeleton and air pores, generates phenol-formaldehyde solid waste. Formaldehyde resin is used to crosslink, or glue, fibers together, so that most of the phenol and formaldehyde released after evaporation is found in wastewater, while the remaining part is generated as solid waste. The paper characterizes solid waste using the following chemical analyses: UV-VIS spectrophotometry, atomic absorption spectrophotometry, and gas chromatography. This is done with the aim to provide a more detailed consideration of the pollution potential. Based on the conducted research, it can be concluded that the drainage water according to the criteria for underground water contains increased amounts of copper, cadmium, lead, chromium, cobalt, nickel and phenol. At the same time, the concentrations of copper, cadmium, nickel and phenol also exceed the values, which is a condition that requires more detailed investigations. The concentration of phenol is getting close to the threshold value (0.050 mg/dm^3), according to which measures are recommended. The paper proposes an eco-technical solution for waste stabilization through supercritical aqueous oxidation of phenol-formaldehyde and metal waste.*

Key words: *phenol-formaldehyde waste, metal waste, supercritical aqueous oxidation, waste stabilization.*

1. INTRODUCTION

Environmental awareness is developed through socially responsible behavior and action due to the well-known facts about environmental pollution, global warming and climate change, as well as the decreasing amount of nonrenewable natural resources due

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to anthropogenic causes. The development of environmental awareness is increasingly striving for waste-free technologies, sustainable production, circular economy, use of waste as raw material, and energy efficiency. The term energy efficiency means a continuous and wide range of measures and actions aimed at reducing the consumption of all types of energy with improved increased living and working comfort and standard [1]. The materials used in construction are numerous and diverse, and as science and technology advance, they are being developed and improved. The future of energy efficiency and environmental sustainability is largely dependent on building materials and their improvement and proper selection, as well as our own environmental protection efforts [2-3]. One of the most useful materials of the modern age is an insulation material of inorganic origin. However, due to its chemical composition, domestic legislation classifies it as a potentially hazardous industrial waste if not adequately stabilized [4]. Accordingly, the paper performs a chemical analysis and presents a possible solution for the valorization of this hazardous industrial waste, its processing, and its reuse [5]. Stabilization was performed by supercritical aqueous oxidation in which a fluid at a temperature above its critical temperature and a pressure above its critical pressure was used as a solvent. Physical-chemical characteristics of supercritical fluid are located between the characteristics of liquids and gases, which makes them effective solvents [6]. Supercritical fluids have a high density close to that of a liquid, which results in a high ability to dissolve. On the other hand, they have high diffusivity and low viscosity, as gases, which contributes to easier penetration into the material and dissolution [6,7].

2. MATERIALS AND METHODS

Phenol, formaldehyde, and certain heavy metals are used as relevant parameters for environmental risk assessment in this study, due to the fact that the metal processing industry used to dispose of its waste at the reference landfill. The samples of drainage water, materials from the hazardous industrial waste landfill, land across the landfill, and land 50 m from the landfill were analyzed in this study. The ecological risk of the reference landfill located on the territory of the Republic of Serbia was calculated by determining the highly or less mobile fractions of the aforementioned pollutants. The Rulebook on allowable quantities of dangerous and hazardous matters in soil and methods for their testing was used to assess the impact on the landfill [8]. The primary environmental risk of the landfill was determined by the extraction of solid samples with distilled water under standardized conditions (imitation of rainwater effect), which is similar to the standard procedure of the US EPA, 1977/222. The term aqueous eluate was adopted in the results for this mobile fraction.

A less mobile fraction for solid samples was chosen in order to gain a more thorough understanding of the pollution potential and to take into account the chemical nature of the waste. This fraction is more tightly bound and thus acts as a reservoir for pollutants. Furthermore, solid residues after leaching with distilled water are subjected to distillation under standardized conditions. The second, more tightly bound fraction of phenol and formaldehyde was released from the samples, and these results were marked as distillate. The more mobile metal fraction was also isolated using standardized nitric acid extraction procedures and labeled as HNO_3-eluate.

The methods of chemical analysis, UV-VIS spectrophotometry, atomic absorption spectrophotometry, and gas chromatography were used in the research. Laboratory data were processed by mathematical and statistical methods in order to obtain square arithmetic means by the method of smallest squares and the method of mean square approximation.

3. RESULTS AND DISCUSSION

Based on the methodology and conducted research, the results of the ecological risk of the reference landfill for hazardous waste are presented through the concentrations of phenol and formaldehyde. Table 1 shows the results of phenol and formaldehyde concentrations in drainage water (1) and solid samples (2-4) and the division between the two factions, i.e. aqueous eluate (easily mobile fraction) and distillate (difficult mobile fraction).

Table 1 Concentrations of phenol and formaldehyde in drainage water and solid samples

Type of sample	Parameter	Total	Aqueous eluate		Distillate	
		(mg/kg)	(mg/kg)	%	(mg/kg)	%
1. Drainage water	Phenol	0.046	-	-	-	-
	Formaldehyde	0.004	-	-	-	-
2. Landfill material	Phenol	1.853	1.810	97.68	0.043	2.32
	Formaldehyde	1.827	0.107	5.86	1.720	94.14
3. Land next to the landfill	Phenol	1.553	1.530	98.52	0.023	1.48
	Formaldehyde	0.580	0.295	50.86	0.285	49.14
4. Land 50 m from the landfill	Phenol	3.608	3.550	98.39	0.058	1.61
	Formaldehyde	0.532	0.319	59.96	0.213	40.04

Table 1 demonstrates that the landfill-derived material contains approximately the same amount of phenol and formaldehyde, about 1.85 mg/kg, and that phenol is significantly more mobile than formaldehyde: 97.7% of phenol and only 5.8% of formaldehyde of the total amount thus defined are leached with water. However, the main amount of formaldehyde can be gradually initiated with water, as shown by the distillation test (94.1%). The results of the test of drainage water, which contains 0.046 mg/l of phenol and 11.5 times less formaldehyde than the initial concentrations of both pollutants in the deposited (initial) material, also indicate higher mobility of phenol. Phenol and formaldehyde accumulate in the soil, as evidenced by their relatively high concentrations in the soil and their initiating material. Phenol shows a higher tendency to accumulate in soil than formaldehyde due to the higher volatility of formaldehyde. Phenol mobility is higher in soil than in formaldehyde, as shown by the distribution between phenol and effluent in the distillate. The amount of phenol that was determined in the soil sample (4) based on the amount in the initiating material is consistent with the substance's tendency of phenol to accumulate in the soil (special chemical composition of the soil, absence of microorganisms that decompose phenols, etc.). Formaldehyde is much more strongly bound in the initiating material than in the soil, but it is easily mobile, as the distribution shows: in the soil (3) it elutes with water about 51%, and in the soil (4) about 60%. Based on the results shown in Table 1, it can be seen that the landfill material is a constant source of easily mobile amounts of phenols and formaldehyde and that these pollutants are transported by water to the environment. Permissible concentrations of organic pollutants in the soil are only slightly standardized. According to the Dutch list of standards 8935/1988, which is accepted by the EEC, the concentration of phenol in the soil of 10 mg/kg obliges us to take measures [9].

Table 2 shows the metal concentration in the drainage water and compares it with the reference values. The data in Table 2 indicate that the content of toxic metals in drainage water is higher than the content in standard groundwater (A), for copper, lead, cadmium, chromium, cobalt and nickel. Criterion (B) is also exceeded in the case of copper, cadmium and nickel. By detailed examination of drainage water (based on criterion B), reliable insight into its long-term environmental risk can be gained. The presence of excess levels of heavy metals in drainage water according to criterion (B) shows that this water, after passing through the landfill waste or contaminated soil under the landfill, elutes a fraction of metals that are easily mobile from these environments, as indicated by solid sample tests. Solid samples (material, landfills and soils) are shown in the table, where the concentration of an easily mobile fraction and a heavier mobile metal fraction is presented.

Table 2 Comparison of metal concentration in drainage water with standard values according to the Dutch list 8935/1988: A - reference value for standard groundwater; B - concentration at which more detailed testing is recommended (EEC Regulation)

Parameter	Concentration (mg/l)	A (mg/l)	B (mg/l)
Cu	0.111	0.015	0.050
Zn	0.086	0.150	0.200
Cd	0.014	0.0015	0.0025
Pb	0.034	0.015	0.050
Cr	0.021	0.001	0.050
Mn	0.089	*	*
Fe	0.290	*	*
Co	0.37	0.020	0.050
Ni	0.068	0.015	0.050
Ca	36.6	-	-
Mg	30.1	-	-

* not standardized

Landfill and soil material contains many toxic elements such as Cu, Zn, Pb, Co, and Ni. Concentrations of Cd and Cr in the extracts were below the detection limit. Part of the ions of these metals is in easily mobile form (aqueous eluate) and that part is gradually eluted by atmospheric precipitation and infiltration through the material. The percentage distribution of the total metal content into two fractions is also presented in Table 3.

Metals such as copper, zinc, and manganese are the most prevalent and easily mobile fractions in landfill material. However, copper, zinc, and lead are present in the soil directly adjacent to the landfill (sample 3), while in the soil 50 meters away (sample 4), metals such as copper, zinc and nickel are most prevalent. The fractions in these metals range from 9 to over 50%.

In all three samples (2, 3, 4), the heavier mobile fraction of metal predominates, except nickel in the soil at 50 m from the landfill. Due to the existence of an easily mobile fraction, it can be noticed that the material of the landfill and soil is a constant source of toxic elements for groundwater, as evidenced by the high content of certain metals in drainage water. Most metals have a higher content of metals in the soil than in the landfill, which can be explained by the fact that soils have a higher capacity for binding metals than landfill materials], which are more firmly bound in the soil. When it comes to the intensity of the ecological and chemical impact of heavy metals from the landfill at the time of sampling, it can be seen from the comparison in Table 4 that the concentration of all metals in both samples is still below the maximum residue level (Overcritical Water Oxidization, OCWO) for standard soil. According to EEC regulations, standard land contains 10% organic matter and 25% clay. Therefore, the conclusion can be reported only by determining the extent to which the examined soil samples deviate from the standard sample. Namely, with the change in the composition of the land, the reference values also change, upwards or downwards.

Table 3 The concentration of easily-moving and difficult-moving metal fractions in solid samples (mg/kg dry matter and% distribution of fractions)

Metal	Type of eluate	Sample 2. Landfill material (%)	Sample 3. Land next to the landfill (%)	Sample 4. Land 50 m from the landfill (%)
Cu	Aquatic HNO$_3$	3.465 (28.6) 8.660 (71.4)	2.440 (16.1) 12.680 (83.9)	4.220 (24.0) 13.360 (76.0)
Zn	Aquatic HNO$_3$	5.32 (21.7) 19.22 (78.3)	4.44 (16.8) 22.03 (83.2)	5.93 (28.2) 15.10 (71.8)
Cd	Aquatic HNO$_3$	< 10 < 10	< 10 < 10	< 10 < 10
Pb	Aquatic HNO$_3$	0.36 (0.4) 83.72 (99.6)	4.44 (9.4) 42.92 (90.6)	1.04 (5.4) 18.04 (94.6)
Cr	Aquatic HNO$_3$	< 0.5 < 0.5	< 0.5 < 0.5	< 0.5 < 0.5
Mn	Aquatic HNO$_3$	3.74 (20.8) 14.21 (79.2)	1.79 (0.2) 1087 (99.8)	18.16 (3.2) 549 (96.8)
Fe	Aquatic HNO$_3$	132.3 (4.5) 2781 (95.5)	81.5 (6.6) 1153 (93.4)	1000.1 (43.7) 1290 (56.3)
Co	Aquatic HNO$_3$	0.100 (5.4) 1.740(94.6)	0.140 (1.8) 7.76 (98.2)	0.305 (6.9) 4.12 (93.1)
Ni	Aquatic HNO$_3$	0.5 0.5	0.5 12.24	2.67 (50.2) 2.65 (49.8)
Ca	Aquatic HNO$_3$	168.6 (3.3) 4940 (96.7)	140.8 (11.7) 1058.5 (88.3)	279.5 (10.5) 2372 (89.5)
Mg	Aquatic HNO$_3$	70.4 -	86.24 (4.6) 1792.5 (95.4)	221.2 (38.2) 358.4 (61.8)

Table 4 Comparison of total metal content in soil samples with the criterion from the Dutch list (EEC) 8935. A - reference value for standard soil; B - concentration at which more detailed monitoring of the content of a given metal is recommended

Metal	A (mg/kg)	B (mg/kg)	Land next to the landfill	Land 50 m from the landfill
			Total concentration	Total concentration
Cu	36	100	15.12	17.58
Zn	140	500	26.47	21.03
Cd	0.8	5	< 10	< 10
Pb	85	150	47.36	19.08
Cr	100	250	< 15	< 15
Co	20	50	7.90	4.42
Ni	35	100	12.24	5.32

Based on the conducted research, the drainage water meets the criteria for groundwater (A) and has higher than average concentrations of copper, cadmium, lead, chromium, cobalt, nickel and phenol. The concentrations of copper, cadmium, nickel and phenol also exceed criterion (B), which is defined as a condition that requires more detailed tests. The phenol concentration approaches the value of criterion (C) (0.050 mg/dm^3), according to which measures are recommended.

The concentration of formaldehyde is not standardized, so analogous conclusions cannot be reported, nevertheless, it is very low. The concentration of other metals in drainage water is below the norms for groundwater, with the exception of cadmium.

In solid samples, a particularly light and difficult-to-move fraction of pollutants was determined. The easily mobile fraction represents that part of the total amount of pollutants that can be directly transferred to the waters passing through the given material. The more difficult-to-move fraction is conditionally mobile (depending on the conditions) and represents the reservoir part of the easily mobile fraction. For phenols in all three solid samples, the easily mobile fraction represents about 98% of the total. For formaldehyde, the easily mobile fraction in landfill material represents only 6%, and in soils 51-61%. For metals, the largest share of the easily mobile fraction is in the material of the landfill for copper, zinc and manganese. The concentration of phenol in both soils exceeds the concentration for the standard soil, so detailed tests are recommended.

4. WASTE STABILIZATION BY SUPERCRITICAL AQUEOUS OXIDATION

The oxidation process presented in this study represents oxidation in an aqueous environment under supercritical conditions, i.e. supercritical aqueous (wet) oxidation. The oxidation process can also be described as a more subtle and efficient variant of ordinary wet air oxidation, which is especially efficient and economical in the processing of hazardous chemical waste with a high content of organic toxic substances.

The process is based on the very specific properties of water in the supercritical region (temperature above 374 ˚C and pressure above 221 bar), in which, thanks to a drastic reduction in the dielectric constant of water, normally insoluble organic compounds become completely soluble in water and thus much more susceptible to oxidation and decomposition with oxygen. The efficiency of this process, expressed as the degree of conversion of organic carbon into CO_2 (Total Organic Carbon, TOC), ranges between 99.9% at 400-450 (C, with a retention time of 5 minutes, and 99.9999% at 600-650 (C, with a reactor retention time of less than 1 minute). In practice, however, the oxidation process is performed at a temperature of 550-600 ˚C and a pressure of 250 bar, and in most cases is an autogenic process (for any hazardous waste with a thermal power greater than 4 MJ/kg). The final products of the process, clean water, carbon dioxide, completely oxidized metals, and inorganic compounds, are completely environmentally safe (closing the pollution cycle). In addition, pure liquid CO_2 is a commercially significant by-product.

The costs of processing waste sludge from central municipal wastewater treatment plants using oxidation in supercritical conditions are important to note because they are approximately twice as low as those associated with wet air oxidation or municipal landfill disposal, and even four times less expensive than those associated with incineration.

Supercritical aqueous oxidation, also known as supercritical aqueous oxidation, is one of the many physical, chemical, and biological processing processes that has received particular attention in the last ten years, especially when it comes to mostly organically polluted waste from chemical and related industries. The technological procedure is based on the specific properties of water that are inherent in it when it is under supercritical conditions, i.e. at temperatures above 374 ˚C and under pressure above 221 bar [10-11]. Under these conditions, namely, its dielectric constant, which at 0 ˚C has a value of 87.7, drastically decreases to only 6, and at 600 ˚C even to 2, which makes water a good solvent for organic compounds and oily substances that are normally hydrophobic.

Bringing water under supercritical conditions is caused by the increase of kinetic energy and weakening of hydrogen bonds, also a sharp decrease in its density. Thus, at 374 ˚C it is about 300 kg/m^3, and at 600 ˚C it drops to only about 50 kg/m^3. The viscosity of water in the over-critical (supercritical) area is also significantly reduced, while diffusivity and heat capacity are increased. On the other hand, inorganic salts, under normal conditions mostly soluble in water, are very poorly soluble in the over-critical area. Thus, the solubility of NaCl, which is over 30% at room temperature, decreases from 450 ˚C to only about 100 mg/l, while the decrease in solubility of $CaCl_2$ is even greater and that of about 70% at room temperature decreases to only about 10 mg/l at 450 ˚C.

The process of over-critical wet oxidation is essentially based on the fact that after entering the over-critical area of water, the number of phases suddenly weighs heavier, and reactions above the critical point are very fast, which allows the very efficient decomposition of organic compounds. This process can also be seen as a significantly improved variant of the long-used wet oxidation (WO) process. This oxidation process also takes place in an aqueous environment, but at a lower temperature and under lower pressure than OCWO, i.e. SCWO (Supercritical Water Oxidation) process. A well-mixed mixture of liquid and/or liquid waste, containing organic and/or oxidizable inorganic substances and gaseous phases, usually air, is heated to 150 to 325 ˚C, and under a pressure of 20-200 bar. A well-mixed mixture of liquid and/or liquid waste, containing organic and/or oxidizable inorganic substances and gaseous phases, usually air, is heated to 150 to 325 ˚C, and under a pressure of 20 - 200 bar. The OCWO process has several advantages over the WO process, including the fact that it occurs in the overly critical area of water, is significantly faster, and more efficient, and results in entirely environmentally friendly final oxidation products-some of which even have significant commercial value (liquid CO_2).

Figure 1 shows the technological scheme of the plant for the possible processing of phenol-formaldehyde and metal waste by over-critical wet oxidation (OCWO) or supercritical aqueous oxidation (SCWO).

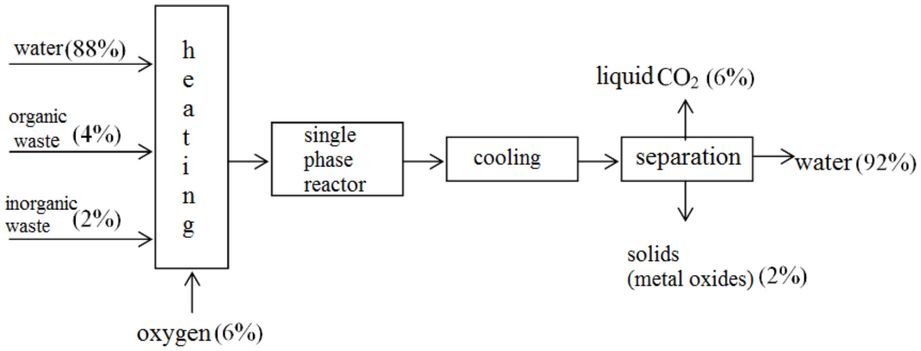

Fig. 1 Technological scheme of phenol-formaldehyde and metal waste processing plants by supercritical water oxidation (SCWO)

Table 5 shows the efficiencies of removal (destruction) of hazardous organic substances using supercritical aqueous oxidation (SCWO) or overcritical wet oxidation (OCWO), with temperature and process time.

Table 5 Results of application of supercritical aqueous oxidation [12-14]

Compounds	Temperature (˚C)	Time (min.)	Efficiency (E%)
Aliphatic hydrocarbons			
Cyclohexane	445	7	99.97
Aromatic hydrocarbons			
Biphenyl	450	7	99.97
Halogenated aliphatic hydrocarbons			
1, 1, 1 - Trichloroethane	495	3.6	99.99
1, 2 - Ethylene dichloride	495	3.6	99.99
1, 1, 2, 2 - Tetrachlorethylene	495	3.6	99.99
Hexachlorocyclohexane	495	3.6	99.99
Halogen aromatic hydrocarbons			
O - Chlorotoluene	495	3.6	99.99
Hexachlorocyclopentadiene	488	3.5	99.99
1, 2, 4 - Trichlorobenzene	495	3.6	99.99
4, 4 - Chlorobiphenyl	500	4.4	99.993
DDT	505	3.7	99.997
PCB 1234	510	3.7	99.99
PCB 1254	510	3.7	99.99
Organic oxygen compounds			
Methyl ethyl ketone	460	3.2	99.96
Methyl ethyl ketone	505	3.7	99.993
Dextrose	440	7	99.6
Organic nitrogen compounds			
2, 4 - Dinitrotoluene	457	0.5	99.7
2, 4 - Dinitrotoluene	513	0.5	99.992
2, 4 - Dinitrotoluene	574	0.5	99.9998

The reason for the slower implementation of this technology in industrial production processes is the sophistication and high investment costs of equipment, as well as the need for a more complex engineering approach, given that relatively high pressures are used during the process [15-16]. Processing of phenol-formaldehyde and metal waste by supercritical aqueous oxidation has a number of advantages because it allows [17-19]:

- control of supercritical fluid power by changing pressure and/or temperature;
- separation of the extract from the supercritical fluid easily and quickly due to the possibility of regulating the volatility of the components by changing the pressure and temperature;
- use of CO_2 which as an agent is environmentally friendly, physiologically safe, nontoxic, nonflammable, and does not cause problems in process engineering;
- supercritical fluid oxidation of plants of different capacities, from analytical (less than grams to several milligrams of the sample), preparative (several hundred grams of the sample), semi-industrial (kilogram of the sample) to large industrial plants (tons of raw materials).

5. CONCLUSION

Based on the methodology and conducted research, the results of the ecological risk of the reference landfill for hazardous waste are presented through the concentrations of phenol and formaldehyde. The paper presents the results of the concentration of phenol, formaldehyde and metals in drainage water and solid samples. On the other hand, the stabilization of hazardous waste by supercritical water oxidation was also carried out. Supercritical aqueous oxidation (over critical wet oxidation) is the most promising destructive process for hazardous waste processing because it has a wide range of potential applications to different types of hazardous waste, it has the ability to be implemented almost always as an autogenic oxidation process and does not produce any environmentally harmful end products. Moreover, even the production of commercially significant liquid CO_2 and its applicability to various types of solid waste all contribute to its potential. This assessment is certainly supported by the relatively low operational costs of the process, as well as the extremely intensive research activity on its further technical improvement and increase of its economic potential.

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REKUPERACIJA FENOL-FORMALDEHIDNOG ČVRSTOG OTPADA NASTALOG U TEHNOLOŠKOM PROCESU PROIZVODNJE NEORGANSKIH TERMOIZOLACIONIH MATERIJALA

Proizvodnja termoizolacionih materijala neorganskog porekla vlaknaste strukture, koji se sastoji od vlaknastog skeleta i vazdušnih pora, za posledicu ima generisanje čvrstog otpad fenol-formaldehidnog porekla. Umrežavanje, odnosno slepljivanje vlakana vrši se formaldehidnom smolom. Veći deo fenola i formaldehida završi u otpadnim vodama, dok se deo generiše kao čvrst otpad. U radu je izvršena karakterizacija otpada sledećim metodama hemijske analize: UV-VIS spektrofotometrija, atomsko-apsorpciona spektrofotometrija i gasna hromatografija. Na osnovu dobijenih rezultata, u radu je prikazano i eko-tehničko rešenje stabilizacije otpada preradom fenol-formaldehidnog i metalnog otpada superkritičnom vodenom oksidacijom.

Ključne reči: *fenol-formaldehidni otpad, metalni otpad, superktitična vodena oksidacija, stabilizacija otpada.*