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QUALITY ASSESSMENT OF VEGETABLE OIL EFFLUENT DISCHARGED INTO SAVA RIVER

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ABSTRACT

Subject of this paper is the analysis of technological waste water within the business complex of BIMAL d.d.Brčko, which is generated as a result of technological processes within the oil refinery, press plant and extraction plant. This paper is aimed at establishing whether the registered values of the tested parameters, based on physicochemical analyses of wastewater samples from different plants of the subject oil refinery, are within the permissible limits, i.e. whether they have a negative impact on the water quality of the Sava River as a natural water recipient and on the entire environment. The analysis comprised 11 parameters, and deviations from national standards for wastewater were identified for one parameter for the sulphate content, which indicates the need for further monitoring and taking adequate measures to prevent adverse environmental impacts.

Key words: *waste water, oil refinery, impact, environment*

INTRODUCTION

Any water that has changed its physical, chemical and biological characteristics after use and is not suitable for its formal or any other use can be considered as waste water. The composition of industrial wastewater depends on the production process and may contain non-degradable as well as biodegradable ingredients. Vegetable oil processing plants generate a large amount of wastewater, which can cause serious environmental problems [1]. The wastewater varies both in its quantity and characteristics from one oil industry to another.

The composition of wastewater from the same industry also varies greatly from day to day, and these fluctuations can also be attributed to different types of processed oils [2]. Vegetable oil industry wastewaters contain a large amount of organic compounds along with high concentration of oil composites [3]. Its characteristics depend largely on the type of oil processed and, on the process, implemented that are high in chemical oxygen demand (COD), oil and grease, sulphate and phosphate content, resulting in both high inorganic as well as organic loading of the relevant wastewater treatment works [4]. The process of sunflower oil production yields free fatty acids that results in acidic and oily wastewater [5]. If the effluent is discharged untreated, it can certainly cause serious environmental problems due to its high biochemical oxygen demand (BOD) and chemical oxygen demand (COD) [6].

Wastewater may also have a high content of suspended solids, organic nitrogen, and oil and fat, and may contain pesticide residues from the treatment of the raw materials [7]. Most of the effluent is generated during refining and the washing process after neutralization and the discharge of poor-quality effluent by vegetable oil industries poses a threat to water resources. The other significant source of wastewater comes from the cooling towers and the acidulation of soap stock with sulphuric acid [8]. According to Jail, A. et al. [9] high phosphorus levels may also be found, in particular when large quantities of phosphoric acid are used in the degumming process of vegetable oils, or in the cleaning. Therefore, the removal of waste oil and other by-products from wastewater from oil-producing factories is an important measure for prevention of the water pollution as well as the environment.

The removal of pollutants by physicochemical and biological methods depends on several factors, such as the concentration of pollutants, their properties and nature, as well as the functionality of the purification devices themselves. As a result, the performance of the purification process varies significantly from case to case. [10]. Because of quantity, characteristic variations and complexity of wastewater it is demanding for the treatment to meet the desired effluent standards, and the choice of methods of waste-water treatment depends on many local conditions and, therefore, cannot be standardized [11]. Previously, effluent from the vegetable oil industry used to be discharged directly into soil or groundwater. But, due to the emergence of environmental consciousness, the Pollution Control Boards have become stricter and imposed stringent norms [12,13].

The extent to which wastewater is to be treated depends on its composition, mass, class and size of recipient and legislation. The basic requirement is that the discharged polluted water does not change the recipient's water quality. Therefore, the goal of a wastewater treatment plant, like any other, is to reduce pollution levels before discharging into the environment to avoid inflicting damages to the environmental balance. New environmental regulations and the increasing market demands for "green" companies are forcing industry to use adequate wastewater treatment methods, as well as regular analysis of water quality after treatment, in order to reduce their negative impact on the quality of water recipients.

The regulation of the discharge of hazardous and harmful substances into natural waters must comply with the Law on Waters, the Decree on water classification and watercourse categorisation, as well as all other regulations for environmental protection, regulations for wastewater and treatment plants, where local conditions in the recipient should be considered in technological, environmental and economic terms. Given the increasing emphasis on environmental pollution control, new laws and regulations and changes in economic factors, the observed oil refinery (BIMAL) has installed a primary wastewater treatment plant, as well as a grease and oil separator in the extraction plant and a multi-chamber separator from the press plant. This company processes 120,000 tonnes of oilseeds (sunflower, soybean and rapeseed) per year. The aim of this study is to evaluate the quality of the wastewater after some treatment in relation to national standards for wastewater and to determine the potential pollution of the water recipient, i.e. of the river Sava.

MATERIALS AND METHODS

Wastewater sampling

After inspecting the production facilities and processes at the subject oil refinery, measurement points for wastewater sampling were determined in order to gain a better insight into the characteristics of the effluent and its potential impact on the water recipient. The wastewater analysis covers the basic groups of physicochemical and chemical parameters defined by the current Rulebook on conditions for wastewater discharge into the public sewage system [14]. Subject sampling included the analysis of technological wastewater within the business complex of BIMAL d.d.Brčko generated as a result of technological processes within the oil refinery and extraction plant, notably:

- Sample I - waste water - effluent discharged from a primary wastewater treatment plant in the refinery and bottling plant
- Sample II – effluent - physically treated waste water at the discharge from the grease and oil separator in the extraction plant
- Sample III - Waste water from the press plant at the outlet of the multi-chamber separator, i.e. effluent entering the sewer system

The listed effluents are discharged into the public sewage system. A primary waste water treatment plant with a capacity of 10,000 l/h was constructed for the treatment of technological waste water generated in the refinery and bottling plant, as well as a separate treatment plant for the treatment of waste water discharged from the press and extraction plant. The treated waste water is further conveyed through inspection chambers and pipelines to the public sewage system, from which point it is conveyed to the natural water recipient - the Sava river, which is classified in Category 2 in accordance with the Decree on water classification and watercourse categorisation [15], it is to say the ecological quality of water.

The aim of this study was to analyse the quality of wastewater in relation to national standards for wastewater discharged into public sewage system, and to determine its potential for pollution of the Sava River as a natural recipient of public sewage. Wastewater pollution can impair the natural balance and functionality of the entire existing ecosystem of this river. Aspects of physicochemical characteristics were studied from 2014 to 2019 in 2 - 4 samples during the year. Eleven (11) parameters were analysed in all three samples using standard methods and compared with the national wastewater standard.

Statistical analysis

The results obtained from samples analysis were processed using Descriptive Statistic Package of Microsoft Excel in order to obtain some of the standard statistical parameters, such as mean, standard deviation (SD), minimum (min) and maximum (max). One-Sample t-test was used to test for significant difference between the effluent characteristics and National Effluent Standard [15]. Differences in concentration levels obtained for a given parameter along sampling locations were considered significant if calculated p values were < 0.05 .

Pearson correlation analysis with significance levels was conducted in order to determine linear relationships among analyzed parameters for each sampling point. The strength and statistical significance of the relation between two water quality parameters is greater as the correlation coefficient is closer to +1 (perfect positive relationship) or -1 (perfect negative relationship). The relationships are characterized as strong, moderate and weak based on the correlation. The relationship is strong, when it is in the range of 0.8 to 1.0 and -0.8 to -1.0, the relationship is moderate when the values are in the range of 0.5 to 0.8 and -0.5 to -0.8, the relationship is weak when it is in the range of 0.0 to 0.5 and 0.0 to -0.5 [16]. Biodegradability index (BOD/COD) calculated with mean concentrations was used for estimating biodegradability of wastewater samples and for assessing the degree of the potential pollution caused by effluent discharge [17].

RESULTS AND DISCUSSION

The mean values of the tested physicochemical and chemical parameters from the three sampling points, as well as the limit values defined by the Rulebook on conditions for wastewater discharge into the public sewage system [14] are shown in Table 1. Wastewater temperature (T°) differed between the three samples. The lowest temperature was in the effluent discharged from the primary treatment plant (sample I), where the mean value was 22 °C. Slightly warmer was the waste water from sample II (mean value 26.5 °C), while the warmest was waste water from the press plant (sample III) with a mean temperature of 33.6 °C. Effluent temperatures at all three sites varied between samplings, but all recorded values were below the permissible limit and were significantly ($p < 0.05$) different from it. An exception is the effluent temperature from sample III, which in November 2014 and March 2015 equalled to the limit value of 40 °C but did not exceed it.

The measured pH values did not differ significantly between the three sampling sites, where the mean values were about 7.5. All values were within the permissible limit values ($p < 0.05$). The lowest pH of 6.6 in March 2017 and the highest of 9.1 in December 2016 were identified in sample I. pH is an important indicator of water quality because many other chemical processes depend on it. The authors who tested wastewater from a vegetable oil processing plant registered different pH values. Cases where the sample was taken from untreated wastewater registered much lower pH values [18,19]. The main cause of the acidity of effluents from this industry is the addition of sulfuric acid into soap stock to separate free fatty acids from the medium [5]. On the other hand, pH values were higher in the wastewater treated before discharge [8,6,10].

Table 1. Physicochemical characteristics of wastewater effluent from three different sample sites and National effluent standard

Parameters*	Standard analytical methods	Sample I Mean \pm SD (min-max)	Sample II Mean \pm SD (min-max)	Sample III Mean \pm SD (min-max)	National Effluent Standard
Temperature (°C)	BAS DIN 38404-4:2010	22.24 \pm 6.12 (12.3-30.5)	26.5 \pm 4.34 (17.2-34)	33.64 \pm 6.58 (12-40)	40
pH (pH units)	BAS ISO 10523:2013	7.37 \pm 0.57 (6.61-9.17)	7.61 \pm 0.54 (6.7-8.94)	7.58 \pm 0.29 (6.8-8.17)	6,50-9,50
Total Suspended Solids	BAS EN 872:2006	65.1 \pm 112.14 (2-480)	24.16 \pm 20.67 (2-68)	50.55 \pm 101.68 (2-412)	500
Biochemical Oxygen Demand	BAS ISO 5815:2004	28.07 \pm 38.81 (2.83-140)	19.1 \pm 22.19 (0.8-86)	21.94 \pm 22.08 (0.56-67)	-
Chemical Oxygen Demand	BAS ISO 15705:2005	154.17 \pm 246.16 (26.2-1086)	72.48 \pm 38.94 (32.4-89.4)	84.48 \pm 49.77 (17.3-164.6)	-
Total Kjeldahl Nitrogen	BAS ISO 5663:2000	2.10 \pm 2.97 (0.11-14.01)	1.88 \pm 1.39 (0.6-5.6)	1.72 \pm 1.31 (0.56-5.44)	100
Total Phosphorus	BAS EN ISO 6878:2006	2.09 \pm 1.84 (0.01-4.86)	0.76 \pm 0.61 (0.1-2.12)	0.89 \pm 0.9 (0.05-3.7)	5
Oil and Grease	EPA 1664-R-A: 1999	57.16 \pm 56.53 (5-186.4)	30.57 \pm 17.58 (4-62.3)	33.75 \pm 27.4 (6-88.5)	-
Sulphate	ASTM D 516:2007	116.02 \pm 152.42 (0.68-671.9)	15.29 \pm 4.9 (3.49-23.6)	23.75 \pm 19.69 (4.6-77.31)	200
Chloride	ISO 9297:1989	29.57 \pm 32.52 (3.4-96)	12.79 \pm 9.45 (1-33.68)	13.2 \pm 18.06 (3.34-66.2)	250

* All units in mg/l, except pH and temperature

The concentrations of total suspended solids (TSS) were significantly lower ($p < 0.05$) than the limit value at all three measurement locations. The lowest TSS concentration was in sample II (mean value 24.16 mg/l), where the maximum value of 68 mg/l was recorded in November 2014. In sample I (mean value 65.1 mg/l) and sample III (mean value 50.55 mg/l) maximum values reached 480 mg/l in December 2016 and 412 mg/l in October 2017. Similar values were reported by Anyanwu et al. [19] and Khoufi and Aloui [6], while significantly higher concentrations have been reported in untreated wastewater of this type [10,18]. It is at these two measurement sites (sample I and sample III) that there is a positive correlation of the mean grade between TSS and oil and grease (O&G) concentrations (Tables 2 and 4). The correlation between these parameters has also been reported by

other authors [19], and Ikhu-Omoregbe et al. [8] explains this relation through the ability to register an oil as a TSS in a sample depending on its temperature and other physical characteristics.

The precipitants determined in Imhoff tanks were less than 0.2 ml/l in almost all samples. The maximum recorded value of 3.9 ml/l was in sample I in December 2016, which is still below the permissible limit of 5 ml/l. The content of precipitates was not presented in tables due to the manner of presenting the results of their analysis and the inability to calculate statistical parameters, as it was the case with the other tested physicochemical and chemical variables.

Table 2. Pearson's Correlation coefficients among effluent parameters of Sample 1

	T	pH	TSS	BOD	COD	TKN	TP	O&G	SO ₄	Cl
T	1									
pH	-0.301	1								
TSS	0.006	0.667*	1							
BOD	-0.154	0.384	0.713*	1						
COD	0.03	0.265	0.555*	0.919**	1					
TKN	-0.123	0.685*	0.917**	0.53*	0.274	1				
TP	0.575*	0.057	0.326	0.053	0.105	0.321	1			
O&G	-0.234	0.357	0.521*	0.577*	0.318	0.539*	0.114	1		
SO ₄	0.175	0.157	0.489	0.826**	0.946**	0.18	0.228	0.351	1	
Cl	0.606*	-0.099	0.104	-0.04	0.164	-0.067	0.573*	-0.087	0.392	1

* p<0.05; ** p<0.01

Table 3. Pearson's Correlation coefficients among effluent parameters of Sample II

	T	pH	TSS	BOD	COD	TKN	TP	O&G	SO ₄	Cl
T	1									
pH	-0.531*	1								
TSS	0.53*	-0.514	1							
BOD	0.207	-0.168	0.286	1						
COD	0.284	-0.191	0.421	0.858**	1					
TKN	0.024	-0.062	0.083	0.84**	0.627*	1				
TP	0.408	-0.446	0.365	0.114	0.099	0.14	1			
O&G	-0.215	0.482	-0.183	-0.497	-0.332	-0.367	-0.224	1		
SO ₄	0.311	-0.196	0.488	0.21	0.223	0.292	0.667*	-0.132	1	
Cl	-0.3	0.628*	-0.4	-0.539*	-0.522*	-0.258	0.022	0.619*	0.193	1

* p<0.05; ** p<0.01

Table 4. Pearson's Correlation coefficients between effluent parameters of sample III

	T	pH	TSS	BOD	COD	TKN	TP	O&G	SO ₄	Cl
T	1									
pH	-0.026	1								
TSS	-0.191	-0.4	1							
BOD	0.428	-0.069	-0.293	1						
COD	0.335	-0.228	0.254	0.646*	1					
TKN	0.168	0.329	-0.166	0.476	0.509	1				
TP	0.014	-0.691*	0.579*	-0.336	0.141	-0.166	1			
O&G	0.004	-0.303	0.666*	-0.533*	0.051	-0.304	0.569*	1		
SO ₄	-0.051	-0.467	0.844**	-0.299	0.284	-0.22	0.563*	0.753**	1	
Cl	-0.099	-0.35	0.237	-0.45	-0.265	-0.26	0.439	0.273	0.197	1

* p<0.05; ** p<0.01

The BOD₅ values were highest in the effluent discharged from the primary treatment plant (sample I), 28.07 mgO₂/l on average, while the mean values in sample II and sample III were slightly lower, 19.1 mgO₂/l and 21.94 mgO₂/l, respectively. The BOD₅ values of sample I ranged from 2.83 mgO₂/l in June 2018 to 140 mgO₂/l in November 2014. The minimum amount of biological oxygen consumption in sample II (0.8 mgO₂/l) and sample III (0.56 mgO₂/l) was recorded in October 2017, while the maximum for sample II (86 mgO₂/l) and sample III (67 mgO₂/l) was in March 2015.

Chemical oxygen consumption (COD) values are consistent with BOD₅. The highest concentrations were registered in the effluent of sample I, an average of 154.17 mgO₂/l, and the minimum (26.2 mgO₂/l) and maximum (1086 mgO₂/l) values were recorded at the same time as BOD₅, in June 2018 and November 2014 respectively. Values at other two sampling points are lower, the mean value in sample II is 72.48 mgO₂/l and in sample III 84.48 mgO₂/l. Maximum concentration in sample II (189.4 mgO₂/l) was registered at the same time as the maximum value of BOD₅, in March 2015, while for sample III the maximum value (164.6 mgO₂/l) was registered in March 2016. Minimum chemical oxygen demand, 32.4 mgO₂/l, from sample II was registered in December 2018, and from sample III, 17.3 mgO₂/l, in June 2019. According to the current Rulebook on conditions for wastewater discharge into the public sewage system: "BOD₅ and COD are not standardised, they are regulated by permit taking into account all the technical and economic factors affecting the selection of a common treatment plant and the penetration of groundwater into the sewer system"[14]. The limit values have not been established for this reason.

It is usually possible to establish a linear interdependence between BOD and COD [20]. The correlation between BOD₅ and COD at all three sampling sites was significant and positive (Tables 2,3 and 4), and the degree of relation was strongest in sample I ($r = 0.919$). BOD₅ also had a positive median (samples I and III) and a strong relation (sample II) with total Kjeldahl nitrogen (TKN). I Anyanwu et al. [19] reported a strong positive correlation (0.843) between BOD and COD as well as between BOD and nitrates. What distinguishes sample I from the other two sampling sites is the strong positive relationship between BOD and sulphate content (SO₄²⁻) ($r = 0.826$), which is absent in samples from the other two measurement locations, as well as a positive median correlation with oil and grease content (O&G) which is negative in sample II and III (Tables 3 and 4).

Just like BOD, COD in sample I was significantly and positively correlated with the sulphate content (SO₄²⁻) (Table 2), while in samples II and III, a mean positive correlation existed with TKN (Tables 3 and 4).

Similar average values of these two parameters (BOD and COD) were registered in the wastewater of this type, which has undergone some type of treatment in other studies [6,18,21], while in untreated or insufficiently treated wastewater [8,17,22,23] concentrations were multiply increased.

The biodegradability index (B.I), defined as the relationship between biological and chemical oxygen demand (BOD/COD), is one of the generally accepted methods for assessing wastewater biodegradability. Its advantage over other indicators of biodegradability is that it depends neither on the quantity nor on the oxidation state of the organic matter [24]. On the other hand, obtaining BOD results requires five days of incubation, and the BOD test itself is often not reliable in the case of low concentrations [25]. The problem may also be the potential presence of heavy metals and other toxic substances in wastewater that, even at low concentrations, can interfere with bacterial activity and thus inhibit biodegradation [20]. In practice, examination of the presence of these toxic substances is often not included in regular monitoring and it is difficult to determine whether low BOD values are only caused by reduced organic load [25].

Wastewater from vegetable oil processing plants most often has a BOD/COD ratio of about 0.2 [2,19]. This is also the case with the wastewater of the business complex of BIMAL d.d. Brčko, where the BOD/COD value in sample I was 0.18, in sample II 0.22, and in sample III 0.24. BOD/COD values <0.2 classify wastewater as biodegradable (sample I) and values between 0.2 and 0.3 in medium degradable (sample II and sample III) [26]. Anyanwu et al. [19] reported a biodegradability index of 0.19, while Verla et al. [18] and Chatoui et al. [23] registered somewhat higher values, 0.5 and 0.3.

The production of vegetable oil generally generates large quantities of waste grease and oils and fats [7,27], and their limit value has not been defined by the aforementioned rulebook. For industrial and other effluents containing toxic or hazardous substances not defined by the rulebook, limit values are determined by a special procedure - based on toxicity, biodegradability resistance, possible carcinogenicity, volume and concentration in the effluent or effluent of the pre-treatment plant, as well as based on international standards regulating the discharge of these substances. In the Federation of Bosnia and Herzegovina, the limit value for the discharge of oil and grease into the public sewage

system is 100 mg/l [28], in the Republic of Serbia 50 mg/l [29], and according to World Bank Group standards 10 mg/l [7].

The oil and grease (O&G) content of sample I effluent had an average of 57.16 mg/l, while their mean values in sample II and sample III were lower by about 40%, 30.57 mg/l and 33.75 mg/l, respectively. The difference between the samples is more significant if the maximum values are compared. In June and October 2016, the oil and grease content in sample I was 186.4 mg/l, while in sample II in March 2018 the value reached 62.3 mg/l and in sample III 88.5 mg/l in December 2017. The studies showed that untreated waste water from the oil refinery contains high concentrations of O&G [8,10,23]. If found in the aquatic environment, oils and greases can cause serious problems, such as reducing light penetration and thereby reducing photosynthesis [30] and impaired oxygen transport from the atmosphere to the aquatic environment, reducing the amount of the dissolved oxygen and endangering the survival of life in the aquatic environment [31].

Like total nitrogen (TKN), total phosphorus (TP) content was highest in effluent from sample I (mean value 2.09 mg/l, maximum 4.86 mg/l in March 2017), while mean values of sample II and III were lower, 0.76 mg/l and 0.89 mg/l. All values were lower and significantly different ($p < 0.05$) from the standard. There is a positive mean degree of relation between TP and sulphate content (SO_4^{2-}) in sample II and III (Table 3.4). Ikhu-Omoregbe et al. [8] and Anyanwu et al. [19] reported similar values, while some authors reported significantly higher concentrations, Verla et al. [18] 890 mg/l, Adakole [21] 2535.87 mg/l. The high concentrations of phosphorus in the wastewater of this industry can be explained by the usage of phosphoric acid in the degumming step process, that is, for the removal of phospholipids and lipoproteins [2].

Sulphate content (SO_4^{2-}) is a nutrient which concentration differs most significantly between sampling sites. The analysed samples from wastewater of sample II vary between 3.49 mg/l in December 2017 and 23.6 mg/l in January 2019, and the water from sample III between 4.6 mg/l in March 2016 and 77.31 mg/l in October 2017. All are lower and significantly different ($p < 0.05$) from the permissible limit value.

However, the concentrations of sulphate (SO_4^{2-}) in the effluent from sample I are higher. The mean value was 116.02 mg/l, and the analysis of the samples showed twice the concentrations above the permissible limit values - 671.9 mg/l in November 2014, which was the maximum recorded value, and 226 mg/l in June 2016. The content of SO_4^{2-} in sample I has a significant strong positive correlation with BOD and COD (Table 2), which is not the case in the other two samples. Verla et al. (2014) reported a mean concentration of 648 mg/l in Port Harcourt, Nigeria [18] and Adakole (2011) as high as 16500.21 mg/l [21], while Anyanwu et al. (2019) registered lower 0.57 mg/l [19].

Chloride content (Cl⁻) follows the trend of other analysed nutrients. The concentrations in the effluent from sample I (mean 29.57 mg/l) are slightly higher than the concentrations in sample II (mean 12.79 mg/l) and sample III (mean 13.2 mg/l). All values are significantly lower ($p < 0.05$) than 250 mg/l - permissible limit values prescribed by the rulebook. Other authors have reported higher chloride (Cl⁻) values in untreated wastewater from vegetable oil refineries, Anyanwu et al. 69 mg/l [19] and Verla et al. 890 mg/l [18]. Ikhu-Omoregbe et al. [8] reported variations from 5.5 to 315.0 mg/l in effluent undergoing primary treatment of purification.

CONCLUSION

Considering the rapid expansion of modern society from industrial and technological aspects, it is necessary to introduce regular monitoring and assessment of the environmental risks of waste materials that are discharged daily. Waste water from vegetable oil processing plants is a complex mixture of organic and inorganic substances. Within the plant for the vegetable oil production "BIMAL" d.d.Brčko, wastewater generated as a result of technological processes within the oil refinery, press plant and extraction plant, after primary treatment is discharged into the public sewage system and from that point into the natural water recipient - the Sava River. Based on the physicochemical analyses of the wastewater samples from the primary treatment plant, the samples

from the grease separator in the extraction plant, and at the discharge of the multi-chamber separator from the press plant, the registered values for the majority of the tested parameters were determined to be within the permissible values.

The sulphate content in two cases in sample I exceeded the permissible values in relation to national standards, and the variation of suspended solids (TSS), temperature, oil and grease (O&G), and biological (BOD) and chemical oxygen demand (COD) were observed. National wastewater regulations have not prescribed permissible limit values for some parameters (BOD₅, COD, fats and oils), preventing the intervention of the competent authorities in order to prevent pollution of the water recipient. The biodegradability index was low, and it is difficult to determine whether the significantly lower BOD values than the COD values were caused by reduced organic load or the presence of some hazardous substances which inhibit microbial degradation. We propose that regular maintenance of the internal drainage system, wastewater treatment and continuous monitoring continue to ensure the satisfying quality of technological wastewater discharged through the sewage system into the Sava River watercourse, in order to prevent the plant from having a negative impact on the quality of the water recipient.

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