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GAMMA-IRRADIATION COMBINED WITH OZONIZATION AS A METHOD OF DECOMPOSITION OF IMPURITIES IN TEXTILE WASTES

Dyebath and washing baths, staple kinds of textile sewage, were treated by the following methods: irradiation, irradiation and aeration, ozonization and simultaneous irradiation and ozonization. The aim of these experiments was to compare the yield of impurity destruction in effluents treated. All basic physicochemical parameters of sewage treated were determined according to the Polish Standards. The best results were obtained in the case of simultaneous use of ozonization and irradiation, which gave the positive synergetic effect. In the investigated range of radiation doses and feed rate of ozone, there was observed a significant reduction of the following effluent parameters: chemical oxygen demand, anionic and nonionic detergent contents and the threshold values of colour and petroleum benzine-extracted substances. The effluents were treated in two stages: ozonization followed by γ-irradiation and inversely, γ-irradiation followed by ozonization. Such a treatment was also effective – purification of dyebath and washing bath proved to be much better than in the case when the above methods were applied separately.

The simultaneous use of ozone and irradiation seems to be promising as the wastewater treatment method on the technical scale.

1. INTRODUCTION

The wastes from textile industry belong to the most noxious for natural environment as they are highly resistant to neutralization. The are usually intensively coloured and contain great amount of detergents, oils, fats and variety of inorganic compounds. Many of these components are toxic to water biocenosis and can hardly be biodegraded. Classical (physicochemical or biological) treatment gives usually poor results and is very complicated and expensive. Hence new methods of wastewater treatment, including radiation techniques, are extensively studied.

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The first papers on applying ionizing radiation to the treatment of sewage were carried out in early seventies and were focused mainly on decolourization of water solutions and decomposition of detergents in some model systems [1]–[7].

In other papers on treatment of model and real wastes, typical chemical methods applied were described [8]–[10]. The optimization of the process [11], [12] as well as different technologies [13], [14] were also studied. Until now, the radiation method of decomposition of impurities in textile effluents has not been applied on the industrial scale. Furthermore, there is a lack of new papers in this area in last years. On the other hand, ionizing radiation attracts much attention as one of the processes in the whole cycle of the treatment of wastewaters, especially municipal ones.

The other process extensively studied recently is the ozonization of wastewaters, also those from textile industry [15]–[20]. The advantages of the latter process are as follows: the lack of secondary pollution of wastewaters by the products of oxidant reduction, easy gaining of raw material (i.e., air), very slight possibility of overdosage and small dimensions of the installation. The main disadvantages are high energy consumption and capital cost.

The combination of both factors, namely ozone and ionizing radiation, was applied by Japanese in order to decompose phenol [21] and ethylene glycol [22] in water solutions. In both cases a positive synergetic effect was observed.

Our paper on the use of ionizing radiation in the treatment of wastes we started with the investigations on the decomposition of typical dyes [23] and detergents [24] in model solutions. Further papers were aimed at the optimization of the process of radiation destruction of impurities in model wastewaters simultaning the most typical technological effluents from washing and dyeing process [25]–[27]. In order to reduce the cost of the method, the combined use of a cheap chemical oxidant and much more expensive ionizing radiation [28] as well as a combination of ionizing and a percolating filter were investigated. The results obtained were compared with those for the real wastewaters from knitting industry.

Bellow we present the results of our investigations on the decomposition of impurities in textile effluents by means of ozone and ionizing radiation. Experiments were carried out on artificial wastewaters, i.e. dyebath and washing bath, which contained typical components at technological concentrations.

The use of artificial wastewaters enabled investigating freshly prepared solutions of always constant initial characteristics and drawing definite conclusions.

2. THE INFLUENCE OF IONIZING RADIATION AND OZONE ON WATER AND DISSOLVED COMPOUNDS

The influence of ionizing radiation on pure water is very well known. During primary and secondary processes various intermediate as well as stable products highly reactive against/towards compounds dissolved in water are formed. The most important among these products are hydrated electron e_{aq}^-, $H^\cdot$ and $OH^\cdot$ radicals, H_3O^+ radical cation and H_2O_2 molecules. Due to their reactivity they can react with solutes causing decomposition. If organic molecules are the solutes, the following scheme of reactions can be drawn

$$RH + H^\cdot \rightarrow R^\cdot + H_2, \tag{1}$$

$$RH + OH^\cdot \rightarrow R^\cdot + H_2O, \tag{2}$$

$$RH + e_{aq}^- \rightarrow RH^- \rightarrow \text{dissociation and neutralization products}, \tag{3}$$

$$R^\cdot + H_2O_2 \rightarrow ROH + OH^\cdot, \tag{4}$$

$$R^\cdot \rightarrow \text{decomposition or dimerization products and in the presence of oxygen}, \tag{5}$$

$$H^\cdot + O_2 \rightarrow HO_2^\cdot, \tag{6}$$

$$R^\cdot + O_2 \rightarrow RO_2^\cdot. \tag{7}$$

In the presence of zone, the following reactions should be included:

$$O_3 + H_2O \rightarrow HO_3^+ + OH^-, \tag{8}$$

$$HO_3^+ + OH^- \rightarrow 2HO_2^\cdot, \tag{9}$$

$$O_3 + HO_2^\cdot \rightarrow OH^\cdot + 2O_2. \tag{10}$$

As can be seen the main reactive species in ozonization process are $OH^\cdot$ and $HO_2^\cdot$ radicals, similarly as in water radiolysis. Thus the following scheme is identical in both cases (ozonization or irradiation):

$$RO_2^\cdot + RH \rightarrow ROOH + R^\cdot, \tag{11}$$

$$2ROOH \rightarrow RO^\cdot + RO_2^\cdot + H_2O, \tag{12}$$

$$ROOH \rightarrow RO^\cdot + OH^\cdot. \tag{13}$$

Final products are formed as a result of recombination of organic radicals:

$$2RO_2^\cdot \rightarrow \text{product}, \tag{14}$$

$$2RO^\cdot \rightarrow \text{product}, \tag{15}$$

$$RO^\cdot + RO_2^\cdot \rightarrow \text{product}. \tag{16}$$

And hence the combined action of ozone and ionizing radiation bases on all the products of ozonolysis and radiolysis. Additionaly it can be enhanced by their reactions with ozone dissolved in water. It is worth noting that ozone is regarded as unreactive towards organic compounds present in water. Japanese scientists have observed positive synergetic effect when ozone and ionizing radiation were applied simultaneously to decompose ethylene glycol and phenol in water solutions [21], [22]. The yield of decomposition was several times higher than when these factors were applied separately.

Simultaneous use of ozone and radiation methods leads to the radiation yields of decomposition equal to 40 and 117 for phenol and ethylene glycol, respectively. These values are considerably higher than this representing the sum of radiation yields of reactive products of water radiolysis which is known to be $G(OH)+G(H)+G(e_{aq}^{-}) = 5.7$. It was concluded that oxygenation of impurities is a chain process which proceeds according to the following scheme:

$$H^{\bullet} + O_2 \rightarrow HO_2^{\bullet}, \tag{17}$$

$$HO_2^{\bullet} + O_2 \rightarrow OH^{\bullet} + 2O_2, \tag{18}$$

$$RH + OH^{\bullet} \rightarrow R^{\bullet} + H_2O, \tag{19}$$

$$R^{\bullet} + O_2 \rightarrow RO_2^{\bullet}, \tag{20}$$

$$RO_2^{\bullet} + O_3 \rightarrow RO^{\bullet} + 2O_2, \tag{21}$$

$$RO^{\bullet} + RH \rightarrow R^{\bullet} + ROH. \tag{22}$$

3. OBJECT AND METHOD

Textile industry produces great amount of wastes containing large variety of impurities, depending on product range, technology and raw materials used in processes. About 80% of all impurities (mainly dyes and detergents) come from dyeing and washing operations. However, these processes need only 40 to 60% of water used by textile plant. Neutralization of textile sewage is additionally complicated by the fact that such processes like dyeing are applied periodically and thus impurities of high concentration and toxicity are introduced periodically.

Because of all the above-mentioned features neutralization of textile sewage can be considered as one the most complex problems in environmental protection.

In our investigations, we used two different artificial wastes of the composition given below:

1. Dyebath
   - acid blue 62 (CI 62045) antraquinone dye – 0.04 g/dm^3,
   - direct yellow 44 (CI 29000) azo dye – 0.03 g/dm^3,
   - direct brown 2 (CI 22311) azo dye – 0.03 g/dm^3,
   - polanol S (anionic detergent) – 0.3 g/dm^3,
   - NaCl – 12.5 g/dm^3.
2. Washing bath
   - pretepon G-extra (anionic detergent) – 0.5 g/dm^3,
   - rokafenol N-6 (nonionic detergent) – 0.5 g/dm^3,
   - calcined soda (Na_2CO_3) – 0.5 g/dm^3.

Both raw and treated sewage were analyzed taking into account the following characteristics:

- specific colour,
- colour threshold (CT),
- pH,
- anionic detergent content (AD),
- nonionic detergent content (ND),
- petroleum benzine extracted substances (PBE),
- chemical oxygen demand (COD),
- biochemical oxygen demand (BOD),
- dry residue (DR),
- soluble substances (SS),
- volatile soluble substances (VSS),
- mineral soluble substances (MSS),
- suspensions (S).

All analyses were done according to the Polish Standards.

4. THE TREATMENT OF DYEBATH AND WASHING BATH WITH OZONE AND γ-IRRADIATION

Washing and dyeing wastes were subjected to the action of ozone and γ-radiation in five modes (ways):

1. γ-irradiation followed by ozonization.
2. Ozonization followed by γ-irradiation.
3. Simultaneous ozonization and γ-irradiation.
4. Ozonization.
5. γ-irradiation of air-saturated wastes.

Irradiation has been carried out in radiation chamber containing Co-60 of total activity of 20 kCi. The doses applied ranged from 1.2 to 25 kGy at the dose rate of 0.33 Gy/s.

The ozonization apparatus consisted of ozonizer with equipment, glass reactor and absorption bulbs. The air was pumped by the membrane pump through two drying columns, flowmeter and bubbler into ozonizer. Glass reactor (1.5 dm^3) equipped with stirrer contained 1 dm^3 of wastewaters. The treatment was carried out at room temperature. In experiments with simultaneous use of ozone and radiation, the reaction vessel was in radiation chamber but the rest of the ozonization apparatus was beyond the radiation fields. Through the appropriate system of valves the stream of gases could be directed to absorption bulbs in order to measure the ozone content in the air at the inlet and outlet of the reactor. The ozone concentration was determined by means of the iodometric method.

Ozonization was carried out at the flow rates of ozone–air mixture equal to 5 and 10 dm^3/h, which resulted in ozone concentrations at inlet of the reactor equal to 28.9 and 36.5 mg/dm^3, respectively.

The process lasted from 30 to 300 minutes. Ozone content ranged from 7.2 to 15.6 mg/dm^3.

5. DISCUSSION

The main results of bath and dyeing waste ozonization are presented in table 1, and these of simultaneous ozonization and irradiation are gatherd in table 2.

Table 1

The influence of ozonization time on the changes of physicochemical characteristics of dyebath and washing bath

Parameter	Units	Composition of raw effluents	Composition of ozonizated effluents					
time	min	–	30	60	90	120	210	300
			Washing bath					
pH		9.7	9.3	9.1	9.0	8.6	8.0	7.7
AD	mg/dm^3	123	115	117	111	105	96	82
ND	mg/dm^3	520	470	410	355	320	210	130
COD	mg O_2/dm^3	1460	1460	1370	1370	1260	1220	1220
BOD	mg O_2/dm^3	28	25	32	30	35	23	50
PBE	mg/dm^3	535	475	455	460	420	310	210
DR	mg/dm^3	1120	1130	1150	1110	1070	960	920
SS	mg/dm^3	1090	1090	1110	1090	1090	930	920
MSS	mg/dm^3	590	580	580	570	590	570	580
S	mg/dm^3	30	40	40	20	50	30	0
			Dye bath					
colour		dark green	light green	light yellow	light yellow	light yellow		
CT		1000	67	50	50	50		
pH		7.5	7.2	7.1	7.4	6.9		
AD	mg/dm^3	142	93	65	50	38		
COD	mg O_2/dm^3	440	435	415	385	345		
BOD	mg O_2/dm^3	47	55	38	60	34		
DR	mg/dm^3	13060	13060	13060	13040	13050		
SS	mg/dm^3	13030	13040	13030	13020	13050		
MSS	mg/dm^3	12820	12820	12810	12800	12820		
S	mg/dm^3	30	20	30	20	10		

Figure 1 shows the changes in the effluent characteristics for the parameters chosen (i.e., COD, AD, ND, CT) after irradiation, ozonization and simultaneous irradiation + ozonization under the same conditions. The values of BOD are more dispersed, so in this case we can observe only tendency, not a precise dependence. The other parameters, i.e., DR, SS, MSS, S, have not been significantly influenced by this method of waste treatment. In some instances, from technological point of view, the simultaneous use of zone and ionizing radiation is difficult, while the treatment with a consecutive application of both factors is rather simple. Thus it was necessary to compare the efficiency of these two alternative methods.

Table 2

Physicochemical characteristics of dyebath and washing bath simultaneously treated with ozone and ionizing radiation

Parameter	Units	Composition of raw effluents	Composition of effluents after simultaneous ozonization and irradiation				
time	min		60	120	180	240	300
			Washing bath				
pH		8.7	8.1	7.9	7.6	7.4	7.7
AD	mg/dm^3	150	118	116	112	108	103
ND	mg/dm^3	565	465	420	365	240	215
COD	$mg\ O_2/dm^3$	1560	1470	1380	1340	1280	1200
BOD	$mg\ O_2/dm^3$	225	85	150	105	160	120
PBE	mg/dm^3	535	475	455	460	420	310
DR	mg/dm^3	1190	1040	1070	1070	1020	1000
SS	mg/dm^3	1180	1040	1040	1050	990	1000
MSS	mg/dm^3	550	480	510	530	490	530
S	mg/dm^3	10	0	30	20	30	0
			Dye bath				
colour		dark green	light green	light yellow	light yellow	light yellow	light yellow
CT		1000	67	50	50	25	25
pH		7.9	7.1	7.0	6.5	5.2	5.0
AD	mg/dm^3	142	71	42	35	12	9
COD	$mg\ O_2/dm^3$	440	395	375	353	300	310
BOD	$mg\ O_2/dm^3$	47	52	62	54	48	65
DR	mg/dm^3	13060	13050	13060	13040	13000	12970
SS	mg/dm^3	13030	13040	13040	13030	12970	12930
MSS	mg/dm^3	12820	12820	12830	12810	12770	12750
S	mg/dm^3	30	10	20	10	30	40

The changes in the chosen parameters of dye and washing wastes after simultaneous and consecutive treating with ozone and γ-radiation are shown in figures 2, 3 and 4.

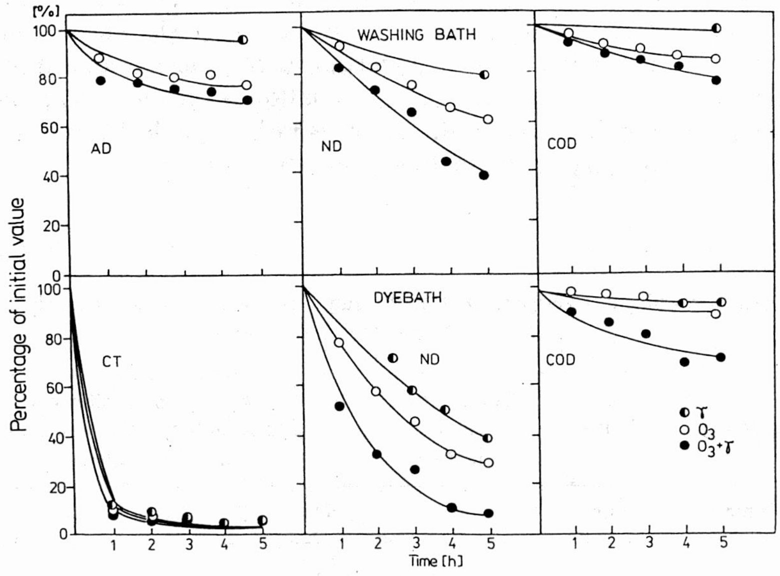

Fig. 1. The influence of the time of irradiation (◐), ozonization (○) and simultaneous irradiation+ozonization (●) on basic physicochemical characteristics of dyebath and washing bath

Dose rate 0.33 Gy/s (120 Krad/h). Ozone feeding rate 145 mg/dm^{3}h

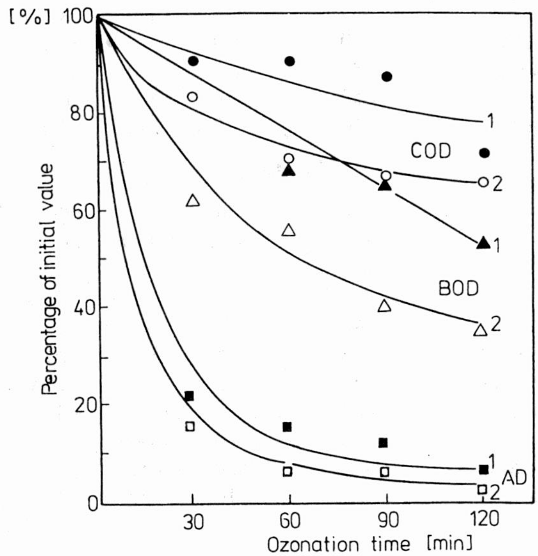

Fig. 2. The comparison of treatment efficiency of dyebath effluents

1 – ionizing radiation followed by ozone, *2* – ozone followed by ionizing radiation.

Total dose 10 kGy (1 Mrad). Dose rate 0.33 Gy/s (120 Krad/h). Ozone feeding rate 365 mg/dm^{3}h

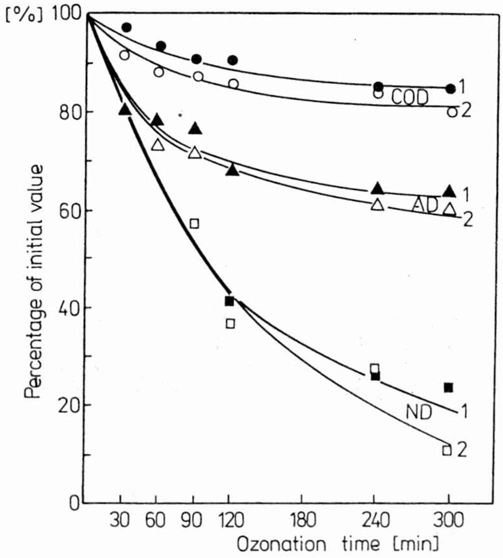

Fig. 3a. The comparison of treatment efficiency of washing bath
1 – ionizing radiation followed by ozone, *2* – ozone followed by ionizing radiation. Total dose 10 kGy (1 Mrad). Dose rate 0.33 Gy/s (120 Krad/h). Ozone feeding rate 365 mg/dm^{3}h

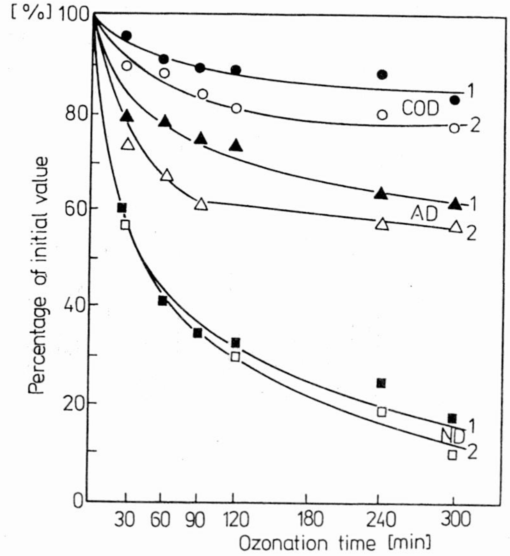

Fig. 3b. The comparison of basic characteristics of washing bath treated with:
1 – ionizing radiation followed by ozone, *2* – ozone followed by ionizing radiation. Total dose 25 kGy (2.5 Mrad). Dose rate 0.33 Gy/s (120 Krad/h). Ozone feeding 365 mg/dm^{3}h

The simultaneous treatment of wastes with ozone and ionizing radiation led to very efficient decomposition of impurities; however, we have obtained better results for dyebath than for washing bath. In the former case, the total decolourization was observed at the radiation dose of 1.2 kGy and ozone concentration of 145 mg/dm^3, while for total decomposition of AD both radiation dose and ozone concentration had to be fivefold higher. Under these conditions COD decreased to 70% of its initial value. Similar results were obtained for washing bath. The degree of detergent decomposition was about 60%.

For both dye and washing wastes the positive synergetic effect is observed, which is cleary seen in figure 1. The results obtained are better than in the case when one of these methods was applied and when the sum of effects of both methods is taken into account (this is true only for low degree of reaction). The increase in the rate and yield of impurity decomposition is probably due to the increase in concentration of very reactive $OH^{\cdot}$ radicals.

The occurrence of positive synergetic effect is of great technological importance because it enables one to lower the dose of ionizing radiation and, in consequence, leads to increase in the yield of the method. This, of course, reduces the cocts of waste treatment.

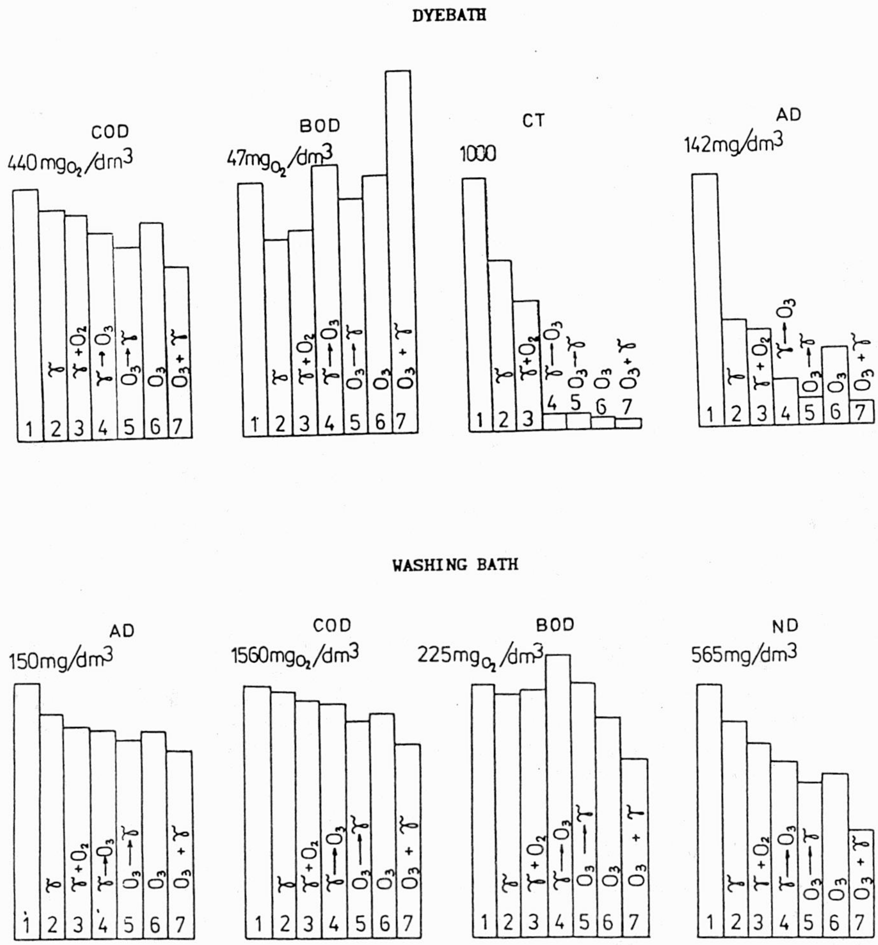

Fig. 4. The influence of treatment method on basic characteristics of dyebath and washing bath
1 – raw, *2* – irradiated, *3* – irradiated and aerated, *4* – irradiated and consecutively ozonizated,
5 – ozonizated and consecutively irradiated, *6* – simultaneously irradiated and ozonizated
Total dose 5 kGy. Dose rate 0.33 Gy/s (120 Krad/h) Ozone concentration 1460 mg/dm^{3}h. Aeration rate 10 dm^3/h

In the case of two-step treatment with ozone and γ-radiation, we have obtained better results when ozonization was followed by irradiation than for the inverse sequence of the treatment. This can better be seen for dye wastes than for washing baths (figures 2 and 3). The difference increases when high doses of ionizing radiation and high concentrations of ozone are used, which is shown in figures 3a and 3b presenting the changes of COD and AD contents in the washing bath analysed.

The change in sewage characteristics after treatment by means of the methods investigated, but at low radiation doses (5 kGy) and low ozone concentration (1460 mg/dm^3), is compiled in figure 4. The effectivenesses of these methods can be presented in the following descending order: simultaneous use of ozone and γ-radiation > ozonization followed by irradiation > ozonization > irradiation followed by ozonization > irradiation.

It is worth noting that BOD increases especially when irradiation is followed by ozonization. The simultaneous decrease in COD evidences that the toxicity of wastes is markedly reduced and thus their biodegradability becomes higher.

Our tests (using three standard microorganisms) on toxicity of effluents prove that COD is five to twenty times lower after irradiation than before [32]. Hence, it is seen that irradiation does not lead to the formation of toxic products as it has earlier been postulated.

The other parameters (i.e., dry residue, dissolved substances and suspended matter) remain unchanged or slightly decreased which has been expected and can be easily explained.

The doses used in our experiments as well as the amount of ozone are too low to decompose impurities into simple molecules like CO_2, H_2O or SO_2. The solid impurities, however, are converted to some extent into more volatile substances, which is supported by significant lowering (up to 200 mg/dm^3 in the case of washing waste) of dry residue.

6. CONCLUSIONS

Radiation technique can be useful in textile waste treatment. The results obtained suggest that simultaneous ozonization and irradiation can be applied successfully on a technological scale. In order to optimize the process of waste treatment, one should take into account purification efficiency as well as total costs of treatment. Thus, such an optimization should be done on the basis of experiments carried out on continuous process, perferentially run in a pilot installation.

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RADIACYJNO-OZONOWA METODA ROZKŁADU ZANIECZYSZCZEŃ W ŚCIEKACH WŁÓKIENNICZYCH

Kąpiel barwiarską i pralniczą, podstawowe rodzaje ścieków włókienniczych, poddano obróbce następującymi sposobami: napromieniowanie, napromieniowanie z napowietrzaniem, ozonowanie oraz jednoczesne napromieniowanie i ozonowanie. Celem doświadczeń było porównanie skuteczności tych metod w rozkładzie zawartych w ściekach zanieczyszczeń. Dokonano oznaczeń wszystkich podstawowych parametrów fizykochemicznych ścieków zgodnie z Polską Normą. Najlepsze rezultaty oczyszczenia ścieku uzyskano, stosując napromieniowanie jednocześnie z ozonowaniem. Stwierdzono w tym przypadku efekt synergistyczny. W przebadanym zakresie dawek promieniowania i ozonu uzyskano wyraźne obniżenie wartości parametrów ścieku, a mianowicie: chemicznego zapotrzebowania tlenu, niejonowych i anionowych środków powierzchniowo czynnych, liczby progowej barwy i substancji ekstrahowanych eterem naftowym. W celach porównawczych przeprowadzono doświadczenia, stosując dwuetapową obróbkę ścieków: najpierw je ozonując, a następnie napromieniowując, i odwrotnie – najpierw poddając napromieniowaniu, a następnie ozonowaniu. Także i w tych przypadkach stwierdzono lepsze rezultaty rozkładu zanieczyszczeń niż wtedy, gdy stosowano obie metody oddzielnie.

Uzyskane wyniki potwierdzają możliwość wykorzystania metody radiacyjno-ozonowej do oczyszczania ścieków włókienniczych w skali przemysłowej.

ОБЛУЧИТЕЛЬНО-ОЗОННЫЙ МЕТОД РАЗЛОЖЕНИЯ ЗАГРЯЗНЕНИЙ В ТЕКСТИЛЬНЫХ СТОЧНЫХ ВОДАХ

Красильная и мойная ванны, основные виды текстильных сточных вод, были подвержены обработке следующими способами: облучение, облучение с аэрацией, озонизация, а также одновременное облучение с озонизацией. Целью экспериментов было сравнение эффективности этих методов в разложении содержащихся в воде загрязнений. Обозначены все основные физико-химические параметры сточных вод согласно Польским Нормам. Самые лучшие результаты очистки сточных вод были получены в случае применения облучения вместе с озонизацией. В этом случае был установлен синергистический эффект. В исследуемых пределах доз облучения и озона получено резкое понижение значений параметров сточнух вод, а именно: ХПК, неионных и анионных поверхностно активных средств, барьерного числа краски и веществ, экстрагируемых петролейным эфиром. Для сравнения проведены эксперименты с применением двухэтапной обработки сточных вод: сначала их озонировали, затем облучали и наоборот – сначала подвергали облучению а затем озонизации. Также и в этих случаях были установлены лучшие результаты разложения загрязнений, чем тогда, когда применяли оба метода отдельно.

Полученные результаты подтверждают возможность использования облучительно-озонного метода для очистки текстильных сточных вод в промышленном масштабе.