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DESTRUCTION OF DYES IN AQUEOUS SOLUTION AND TEXTILE WASTEWATERS BY IRRADIATION

In this paper the attempts of destruction of some dyes in aqueous solutions and textile wastewaters by irradiation method have been shown. Research works were carried out on some selected dyes being the representatives of 4 commercial groups.

Aqueous solutions were irradiated by Co^{60} γ -rays in radiation chamber. The global source activity was 20 000 Ci. The kinetics of the aqueous decolouration solutions of dyes was the matter of research in the range of 0-50 kGy (0-5 Mrad) for several dose rates. The experiments on colour removal from textile wastewater were carried out using artificial baths containing dyestuffs, surface active agents, and other chemicals. The experiments were also performed using the wastewaters oxygenated with pure oxygen. After irradiation the wastewater samples have been submitted to an analytical control of colour, pH, COD, BOD_5 and detergents.

1. INTRODUCTION

In the textile industry large quantities of water are employed which in later stages of treatment carry away the dyestuffs used in technological processes as well as aid agents, chemicals, and the impurities from stock. This causes that textile wastewaters discharged into watercourses contain high loads of organics and inorganics being a mixture of compounds the structure of which is usually very complex. Some of them are biodegradable while the others are nonbiodegradable or toxic toward the microorganisms of the secondary treatment step or they may affect watercourses upsetting the ecology of the receiving waters. Conventional wastewater treatment processes have little or no effect on many pollutants present in textile effluents. Dyestuffs being some of the most resistant materials commonly found in textile effluents are usually responsible for the intensive colour of the textile wastewaters. These refractory substances must be completely removed from the wastewater or their concentration has to be significantly reduced. Dyestuffs discharged

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into textile effluents absorb sunlight, thus limiting the assimilation processes occurring in the receiving waters. According to the available literature the degradation of most dyes in the secondary treatment step is low. In general, the textile dyes are very resistant to biodegradation, and colour removal is less than 50% for most of the wastewaters. That is why proper treatment of textile effluents, including colour removal, can only be achieved by combining chemical or physicochemical and biological methods: a chemical treatment (coagulation) for a satisfactory colour removal should be followed by a secondary biological treatment. However, chemical treatment of textile effluents involves serious problems concerned with the treatment and disposal of post-coagulation sludges. Therefore, it seems to be necessary to develop modern wasteless methods for reduction or removal of refractory materials such as dyestuffs, surface active agents, and heavy metals which are also found in textile effluents. The irradiation of textile wastes is one of the most efficient methods of colour removal. Research works on this method were focussed on colour removal since the organic load of dyes is negligible.

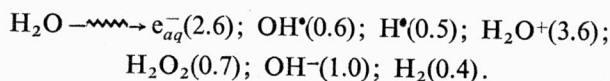
The subject of this work was to conduct preliminary studies on the effect of ionizing radiation on aqueous solutions of selected dyes as well as dye baths used in the textile industry.

2. OUTLINE OF IRRADIATION PROCESS

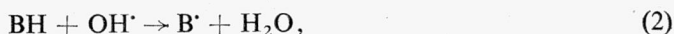
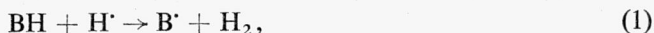
It is well known that a great number of organics are strongly affected by ionizing radiation such as high-energy γ -rays. Organics subject to irradiation undergo various types of reactions including destruction. It has been proved, however, that better results can be achieved by irradiating their aqueous solutions since then primary and secondary products of water radiolysis react with the organic molecules present in water. This process can be improved in the presence of dissolved oxygen or such compounds as: halide ions, rhodanates, and nitrogen monoxide. Usually Cs^{137} , Co^{60} or beam of electrons are as a source of ionizing radiations.

First fundamental studies on the use of irradiation for destruction of dyes or for colour removal from their aqueous solutions have been carried out by HASHIMOTO et al. [5] and SUZUKI et al. [6, 7]. The proposals of using that process for treatment of textile effluents have been also given in other works [1, 2, 4, 8]. It has been suggested that ionizing a solution is more effective if accompanied by the chemical oxidizing agents, such as sodium hypochlorite, hydrogen peroxide, chlor dioxide, and ozone. The detailed works on destruction of dyes deal with acid, azo, and anthraquinone dyestuffs. The destruction mechanism of the tested dyes has been described and some efforts have been undertaken to explain the kinetics of this destruction. In view of the published data it seems that the mechanism of dyes destruction consists of two stages:

1. In the first stage, due to radiolysis of water [7], some primary and secondary products of its destruction may be formed, the yield of radiation is given in brackets:



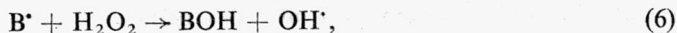
2. In the second stage each of the primary products such as e_{aq}^- , H^\cdot , and OH^\cdot is able to react with organic molecules present in water as well as with dye molecule denoted shortly by BH. Hypothetical interactions are as follows:



The H^\cdot and OH^\cdot radicals can be also attached to dye molecules, viz:

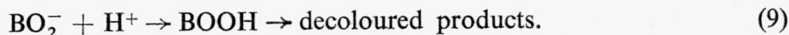
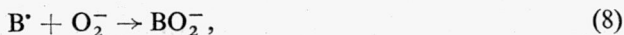


It is also possible that the secondary products of dyes and water radiolysis react in the following way:



thus producing colourless products.

If dissolved oxygen is present in the aqueous solution it may affect the course of reactions. Being a good "sweeper" of hydrated electrons it produces stable anion O_2^- which is very important in oxidation processes, viz:



As it can be seen from the above, the kinetics of colour removal from aqueous solutions of dye depends on the structure of the products formed in consequence of water and dye molecules radiolysis and on their interactions. Yet, if the majority of destruction products are coloured, colour removal processes proceed slowly and are generally not complete.

3. EXPERIMENTAL

3.1. RADIATION SOURCE

Co^{60} was used as a source of γ -rays radiation. This source was housed in an irradiation chamber in the Institute of Applied Radiation Chemistry at the Technical University in Łódź. The irradiation chamber is equipped with 20 radiation Co^{60} sources with total activity of about 20 000 Ci. Dosimetry of γ -radiation was controlled by means of modified Fricke dosimeter [3]. Radiation tests were carried out in a room temperature.

3.2. DYES TESTED

Several dyestuffs were selected as the representatives of the following categories of dyes: direct, acid, and reactive ones. The dyes utilized in the study are listed in tab. 1.

Table 1

Dyes tested
Testowane barwniki

Trade name	Colour index name and number
Helion Yellow G	Direct Yellow 44, 29 000
Polan Blue ERN	Acid Blue 62, 62 045
Prociongrün H-4G	Reactive Green 5, —
Direct Brown RC	Direct Brown 2, 22 311

In addition to trade name, colour index name and numbers are given where available. The chosen dyes represent main groups of dyestuffs used in textile industry. The influence of γ -radiation on destruction of these dyes was investigated taking into account the degree of colour disappearance. Tests were performed using both aqueous solutions of dyes and artificially prepared dyebaths. Aqueous solutions of different dyestuffs were irradiated in the irradiation chamber described above. In the initial phase of this study the aqueous solutions of single commercial dyes were used.

3.3. DYE SOLUTIONS

Taking into account the amounts of dyes in the exhausted dyebaths and in effluents discharged from textile mills, aqueous solutions were prepared with dye concentrations of 0.25 g/dm³, 0.1 g/dm³, and 0.025 g/dm³. Portions of the solutions were transferred into 10 cm³ glass test tubes. Each of them contained a given class of dye at specified concentration. The test tubes were irradiated at radiation doses ranging within 0–50 KGy (0–5 Mrad), dose rates being: 2 Gy/s (720 krad/h), 0.556 Gy/s (200 krad/h), 0.139 Gy/s (50 krad/h), and 0.014 Gy/s (5 krad/h).

3.4. DYEBATHS

Attempts to remove colour from the textile effluents by γ -radiation using artificially prepared dyebaths were also made. The baths were prepared according to technological requirements of dyeing procedure adopted by textile industry. Dyes concentrations in these baths were 0.1 g/dm³. Two dyebaths were prepared. The first one contained direct dye, anionic detergent, acetic acid, and salt. The other one contained reactive dye, anionic detergent, soda ash, and salt. Glass test flasks were filled with 2 dm³ of dyebaths and

then the fixed test flasks were irradiated. Radiation doses were 100 kGy (10 Mrad) and dose rates were 0.278 Gy/s (100 krad/h) and 0.556 Gy/s (200 krad/h). Some of the samples were irradiated at constant oxygen flow rate of 5 dm³/h.

3.5. ANALYTIC CONTROL

The degree of colour removal from dye aqueous solutions was determined at the wavelengths range of 196–800 nm using a spectrophotometer. The range of absorption were 0–0.1, 0–0.5, 0–1, 0–2, and 0–3. Kuvette thicknesses were 2 mm and 10 mm. All measurements were made on Beckman Spectrophotometer Acta M IV.

Analytic examination of dyebaths before and after irradiation included visual inspection of specific colour, determination of the threshold dilution at the moment of colour disappearance, pH, COD, BOD₅, and anionic detergents.

4. RESULTS AND DISCUSSION

4.1. SINGLE DYE SOLUTIONS TREATMENT

Preliminary studies on colour removal from aqueous solutions of the selected dye indicated good colour reduction as a result of dye radiolysis. As it was expected, each of the investigated dyes was characterized by various resistance levels to the ionizing radiation. Figs. 1–4 show the changes in absorption spectra within visual and ultraviolet range under the influence of various radiation doses to individual dyes. The figures illustrate a remarkable disappearance of absorption spectra in visual and ultraviolet ranges with the increasing γ -radiation doses proving the destruction of dye molecules and colour removal.

4.1.1. DYE CONCENTRATION EFFECTS

The studies have shown that total colour removal occurred for the lowest concentration of dye in aqueous solution — 0.025 g/dm³ and at the radiation dose greater than 30 kGy (3 Mrad). The least resistance to irradiation was exhibited by the direct dye Helion Yellow G which was completely destroyed at radiation dose of approximately 10 kGy (1 Mrad). On the other hand, the direct dye (Direct Brown RC) appeared to be most resistant, being destructed at radiation dose of about 30 kGy (3 Mrad). The other dyes (Prociongrün H-4G and Polan Blue ERN) were destroyed at radiation doses ranging within 15–20 kGy (1.5–2 Mrad). At higher dye concentrations (0.1 g/dm³ and 0.25 g/dm³) a total colour removal did not occur even if the radiation dose was increased to 50 kGy (5 Mrad).

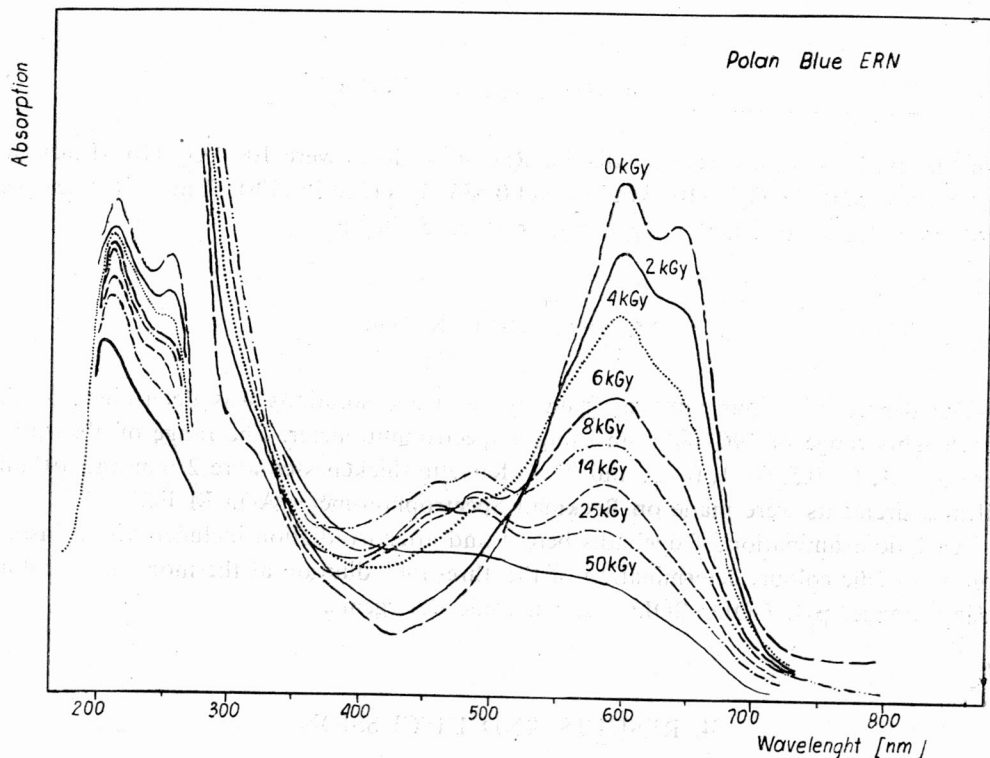


Fig. 1. Absorption spectra of irradiated Polan Blue ERN aqueous solutions
 Rys. 1. Widma absorpcyjne napromienionych roztworów wodnych Polan Blue ERN

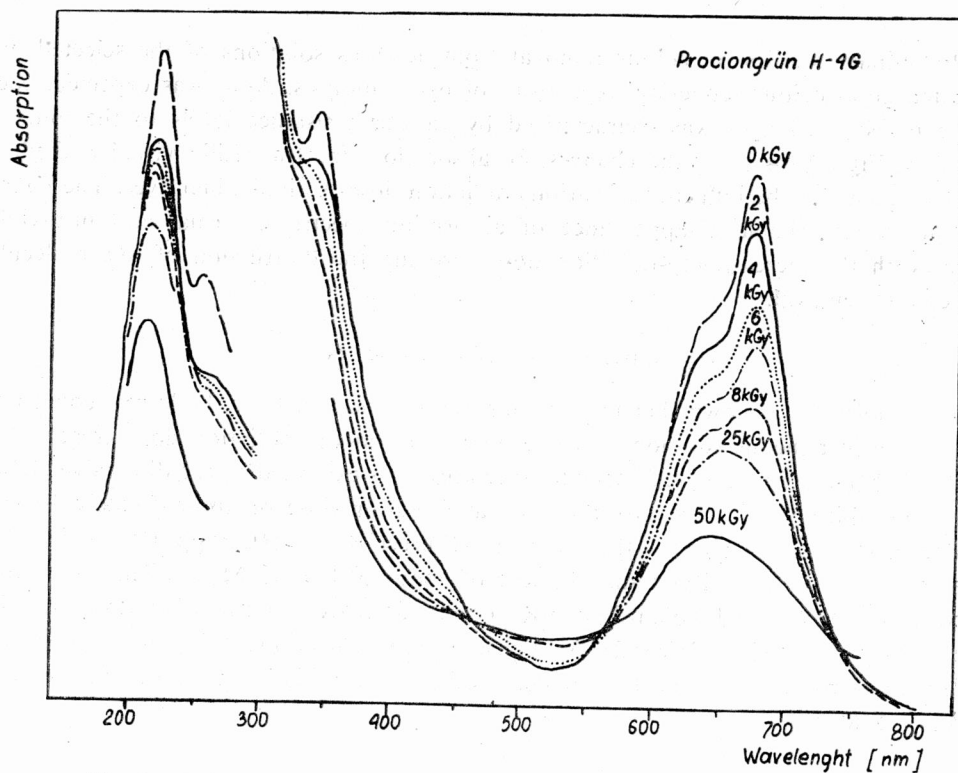


Fig. 2. Absorption spectra of irradiated Prociongrün H-4G aqueous solutions
 Rys. 2. Widma absorpcyjne napromienionych roztworów wodnych Prociongrün H-4G

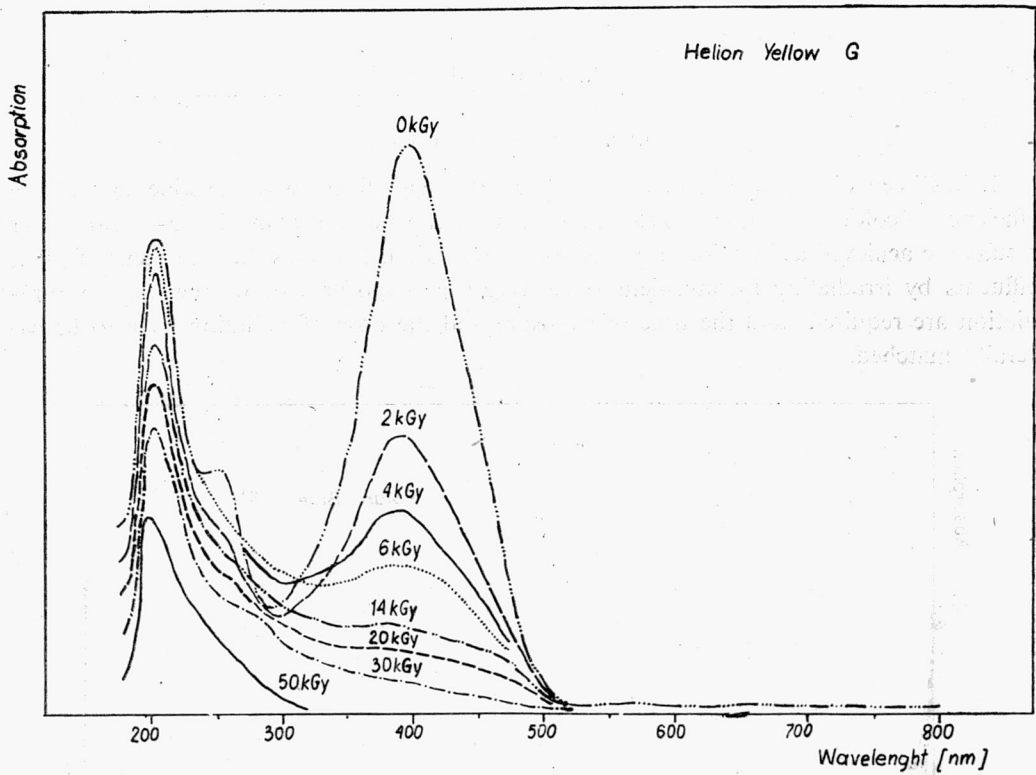


Fig. 3. Absorption spectra of irradiated Helion Yellow G aqueous solutions
 Rys. 3. Widma absorpcyjne napromienionych roztworów wodnych Helion Yellow G

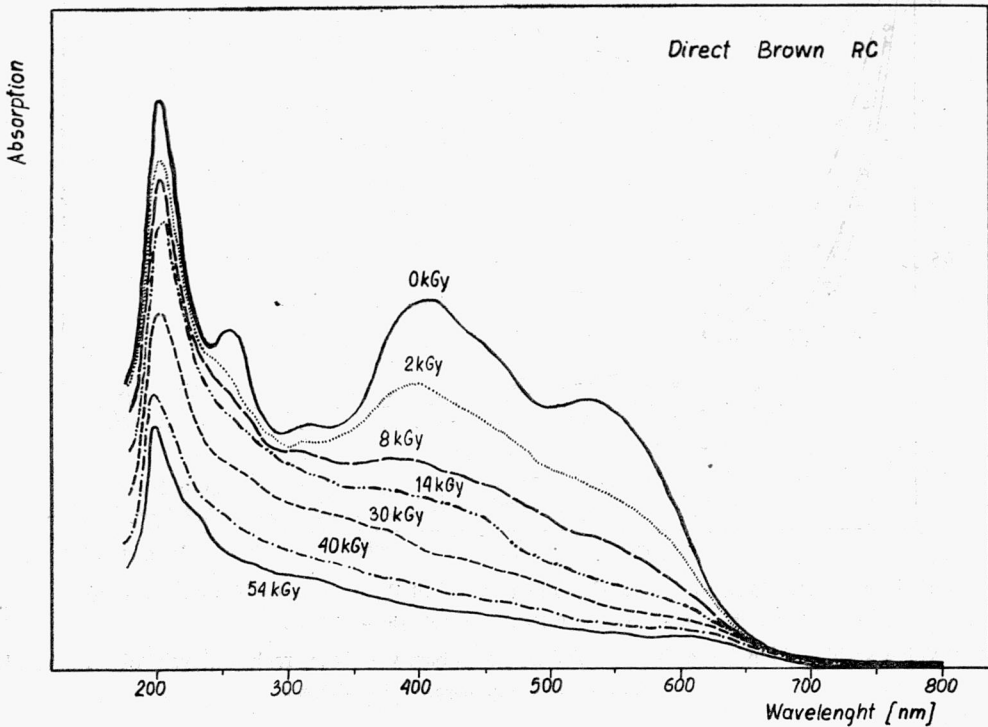


Fig. 4. Absorption spectra of irradiated Direct Brown RC aqueous solutions
 Rys. 4. Widma absorpcyjne napromienionych roztworów wodnych Direct Brown RC

4.1.2. DOSE RATE EFFECTS

It has been observed that dose rate affects in a significant way the kinetics and the efficiency of colour reduction in dye solutions. As it can be seen from figs. 5–6 far better results are achieved at low dose rates of γ -rays radiation. It makes the treatment of plant effluents by irradiating inconvenient since long time exposures of wastewaters to γ -radiation are required; and the time of exposure and the dose of radiation have to be carefully matched.

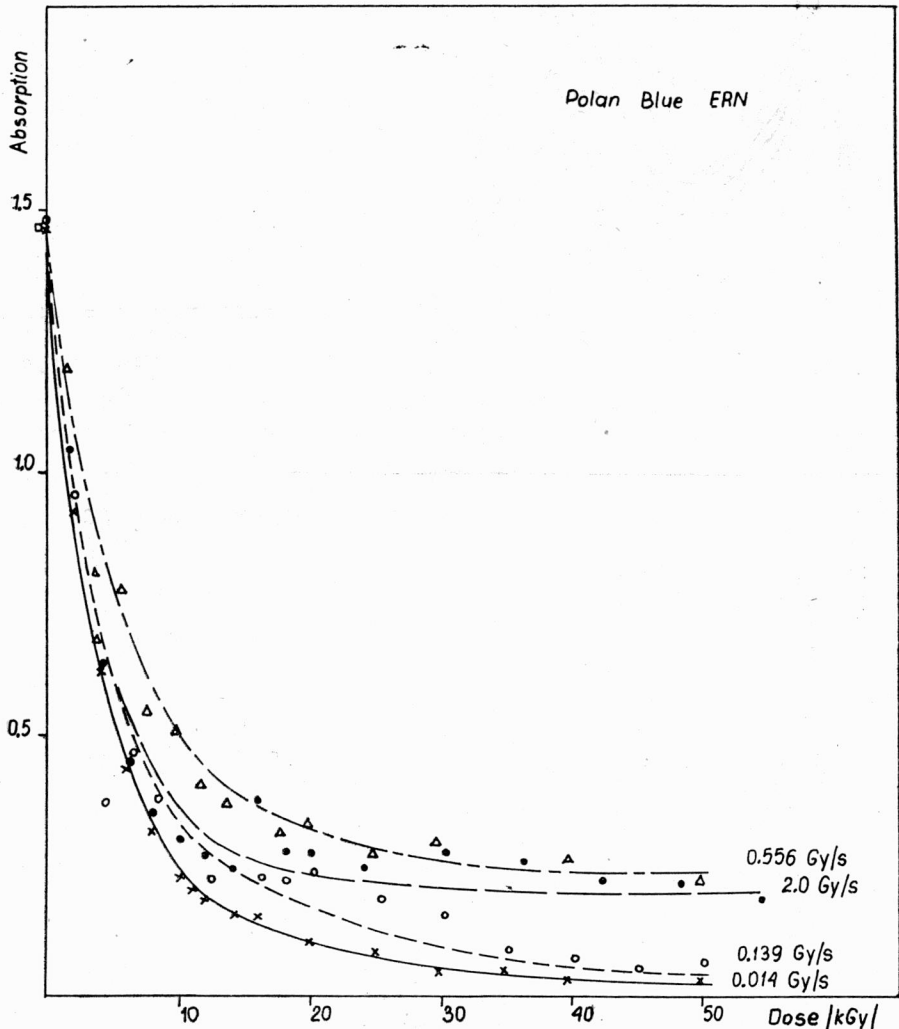


Fig. 5. Dose rate effect on degree of colour removal of Polan Blue ERN aqueous solution
dye concentration – 0.1 g/dm³, wavelength – 640 nm

Rys. 5. Wpływ szybkości dawkowania na stopień usuwania barwy w roztworach wodnych Polan Blue ERN
stężenie barwnika – 0,1 g/dm³, długość fali – 640 nm

4.2. DYEBATH TREATMENT

The dyebaths treated with ionizing γ -radiation belonged to the group of highly concentrated wastes. They were characterized by intensive colour and a heavy load of organics (COD equal to 2 000–2 500 mg O_2/dm^3) and a great amount of anionic detergents (300 mg/ dm^3). The dyebaths had high TDS* concentration.

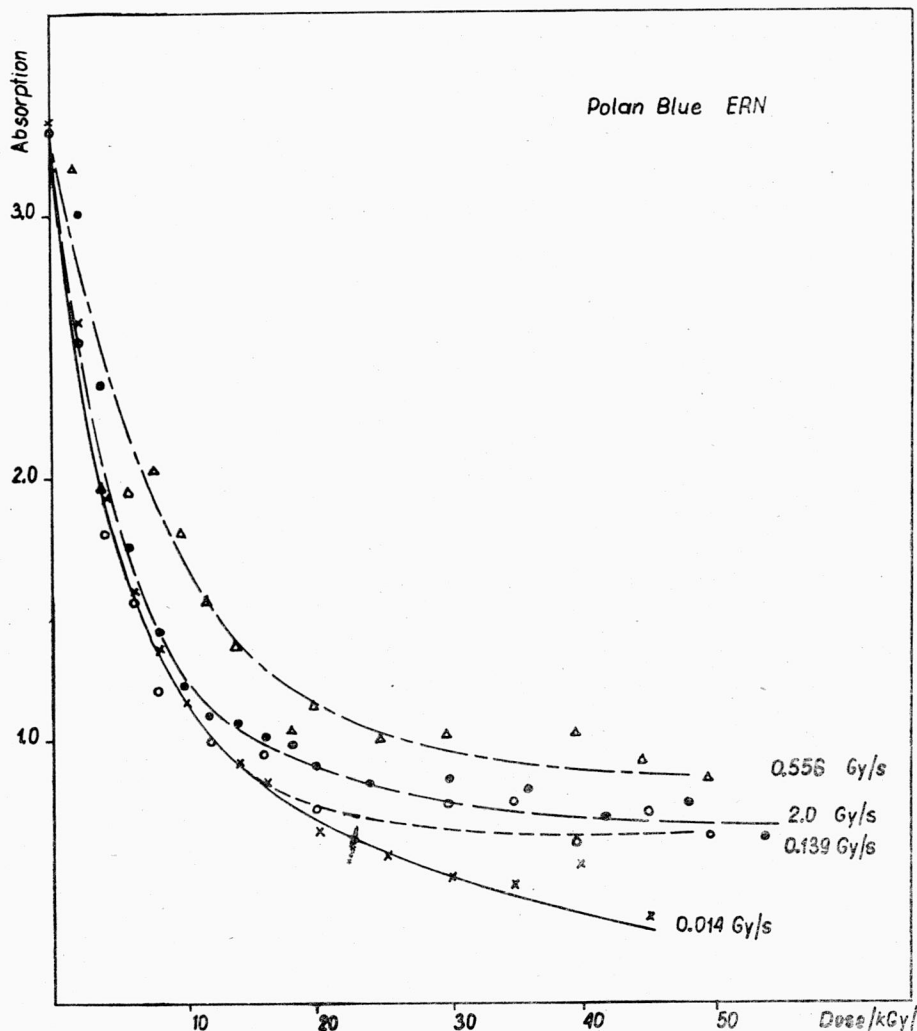


Fig. 6. Dose rate effect on degree of colour removal of Polan Blue ERN aqueous solution
dye concentration — 0.25 g/ dm^3 , wavelength — 640 nm

Rys. 6. Wpływ szybkości dawkowania na stopień usunięcia barwy w roztworach wodnych Polan Blue ERN
stężenie barwnika — 0,25 g/ dm^3 , długość fali — 640 nm

* TDS — total dissolved solids.

Table 2

Irradiation of dyebaths in the absence of dissolved oxygen
 Napromienienie kąpeli barwiących nie zawierających rozpuszczonego tlenu

Parameters	Dyebath containing Direct Brown RC		Dyebath containing Prociongrün H-4G		Dyebath containing Helion Yellow G		Dyebath containing Polan Blue ERN	
	Before radiation	After radiation	Before radiation	After radiation	Before radiation	After radiation	Before radiation	After radiation
Dose (kGy)		100		100		100		100
Dose rate (Gy/s)		0.278		0.278		0.278		0.278
Specific colour	dark-red	light-yellow	green	light-yellow	yellow	light-yellow	dark-blue	light-yellow
Threshold dilution of dyebath to colour disappearance	5 000	50	2 500	330	1 700	170	6 700	33
pH	4.7	5.3	9.3	8.9	9.4	9.0	4.7	5.1
COD (mg O ₂ /dm ³)	2 480	1 200	2 680	1 270	2 780	1 360	2 380	1 950
BOD ₅ (mg O ₂ /dm ³)	430	300	550	280	570	340	400	425
Degree of biological degradation measured as ratio BOD ₅ /COD (%)	17.3	25.0	20.5	21.8	20.4	25.1	16.8	21.8
Anionic detergents (mg/dm ³)	380	50	360	250	370	180	380	73

4.2.1. TREATMENT IN THE ABSENCE OF DISSLOVED OXYGEN

Tab. 2 shows the results of dyebaths irradiation. As it can be seen γ -rays destroyed the dyes in investigated systems. This is confirmed by the fact that after irradiation the wastes became yellowish with dark-brown tint, irrespective of their original colour. Reduction of colour was very high and reached 87–99%. The best results were obtained for wastes containing Polan Blue ERN and Direct Brown RC. From the analysis of results it is evident that beside the destruction of dye molecules γ -radiation caused changes in other organic substances present in dyebaths. These changes were manifested in reduction of such parameters as COD, BOD₅, and anionic detergents. Different levels of reduction of individual parameters can be attributed to different compositions of dyebaths containing various amounts of aid agents and chemicals. It can also be explained by pH, the degree of dyebath mineralization, and dose rate of γ -radiation.

4.2.2. TREATMENT IN THE PRESENCE OF DISSOLVED OXYGEN

The presence of dissolved oxygen in irradiated systems had a considerable influence on reactions of radiolysis. That is why in order to determine the influence of oxygen on irradiated wastes some samples were irradiated at constant flow of pure gaseous oxygen. Irradiation effect on dyebaths in the presence of oxygen is shown in tab. 3. After irradiation the treated wastes became yellowish and the degree of colour removal ranged between 93–99%. In the case of the dyebaths containing Direct Brown RC and Polan Blue ERN the effect of colour removal was similar to that for nonaerated wastes, while in the dyebaths containing Helion Yellow G and Prociongrün H-4G the colour removal was increased by some percent in comparison with nonaerated wastes. Although wastes were irradiated with a high dose at which practically total destruction of dyes molecules occurred, an advantageous influence of oxygen on wastes treatment was still observed. It was clearly seen, particularly in relation to other impurities, in wastes measured by COD, BOD₅, and anionic detergents content.

4.3. EFFECT OF IRRADIATION ON COD OF DYEBATHS

As a result of irradiation of dyebaths in the absence of dissolved oxygen, reduction of COD by 51–53% was obtained, except in the dyebath containing Polan Blue ERN where COD decreased by 18% (tab. 2). Better results were obtained during irradiation of aerated wastes. The range of COD reduction for the dyebath containing Polan Blue ERN was 55%; for the dyebath containing Prociongrün H-4G it was 65%, and for the dyebaths containing Direct Brown RC and Helion Yellow G it amounted to 73% (tab. 3). The results obtained indicate that the destruction process of organic substances occurs in wastes and the effect of their radiolysis is extremely intensified in the presence of oxygen.

4.4. EFFECT OF IRRADIATION ON BOD₅ OF DYEBATHS

As a result of waste irradiation the reduction of BOD₅ ranged between 30–50%. An increase of BOD₅ value by 6% was reported exceptionally in the case of dyebath containing Polan Blue ERN during irradiation of nonaerated wastes. It can be explained

Table 3

Irradiation of dyebaths in the presence of dissolved oxygen
 Napromienienie kąpeli barwiących zawierających rozpuszczony tlen

Parameters	Dye bath containing Direct Brown RC		Dye bath containing ● Prociongrün H-4G		Dye bath containing Helion Yellow G		Dye bath containing Polan Blue ERN	
	Before radiation	After radiation	Before radiation	After radiation	Before radiation	After radiation	Before radiation	After radiation
Dose (kGy)		100		100		100		100
Dose rate (Gy/s)		0.556		0.556		0.556		0.556
Oxygen flow rate (dm ³ /h)		5		5		5		5
Specific colour	dark-red	light-orange	green	light-yellow	yellow	light-yellow	dark-blue	light-yellow
Threshold dilution of dye bath to colour disappearance	5 000	67	2 500	170	1 700	67	6 700	12
pH	4.7	4.4	9.3	7.5	9.4	7.4	4.7	4.3
COD (mg O ₂ /dm ³)	2 480	660	2 680	940	2 780	760	2 380	1 070
BOD ₅ (mg O ₂ /dm ³)	430	230	550	325	570	290	400	280
Degree of biological degradation measured as ratio BOD ₅ /COD (%)	17.3	35.0	20.5	34.5	20.4	38.2	16.8	26.0
Antionic detergents (mg/dm ³)	380	6	360	145	370	120	380	10

by formation of biodegradable compounds. The changes in organic substances due to irradiation of wastes affected also biodegradability of wastes. Throughout the experiments an overall increase of waste biodegradability, measured by BOD₅/COD ratio, was reported. That increase, particularly visible during irradiation of wastes in the presence of oxygen, was by 1.5 to 2 times greater than in nonirradiated wastes. In conclusion, ionizing γ -radiation led to conversion of organic substances into simpler forms which are more suited to biodegradation. The presence of oxygen markedly improved these processes making waste treatment more efficient.

4.5. EFFECT OF IRRADIATION ON ANIONIC DETERGENTS IN THE DYEBATHS

Ionizing γ -radiation led also to removal of anionic detergents in the dyebaths. Effects obtained for wastes irradiated in the absence of oxygen were different and varied between 31–87%. The lowest degree of removal was obtained for the dyebath containing Prociongrün H-4G, the highest one for the dyebath containing Direct Brown RC (tab. 2). Better results were obtained by irradiating dyebaths in the presence of oxygen. The anionic detergents in the dyebaths containing Direct Brown RC and Polan Blue ERN were destroyed up to about 98%. For the other two dyebaths containing Helion Yellow G and Prociongrün H-4G the degrees of removal were 68% and 59%, respectively (tab. 3).

It may be concluded that γ -irradiation results in destruction of the anionic detergents present in the wastes from textile mills and that their radiolysis is highly accelerated by the presence of dissolved oxygen.

5. CONCLUSIONS

As it has been shown γ -rays emitted by Co⁶⁰ cause destruction of dyes in aqueous solutions as well as in textile effluents, leading eventually to the disappearance of colour effect. The degree of colour removal in aqueous solutions and wastes obtained by irradiation ranged within 93–99%. At the same time the irradiation-induced reduction of concentration of organic substances present in wastes was also observed, being stated by measurements of COD, BOD₅, and anionic detergents content. Additionally, the intermediate and final products of the destruction of organic substances due to irradiation were more susceptible to biodegradation than original organic matter.

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RADIOLITYCZNY ROZKŁAD BARWNIKÓW W ROZTWORACH WODNYCH I ŚCIEKACH Z ZAKŁADÓW PRZEMYSŁU WŁÓKIENNICZEGO

W pracy przedstawiono próby radiolitycznego rozkładu niektórych barwników w roztworach wodnych i ściekach z zakładów przemysłu włókienniczego. Badania wykonano na kilku wybranych barwnikach, reprezentujących 4 grupy handlowe.

Roztwory wodne napromieniowywano w komorze radiacyjnej promieniami γ z Co⁶⁰. Całkowita aktywność źródła promieniowania wynosiła 20 000 Ci. Kinetykę odbarwienia barwników w roztworze wodnym badano w zakresie 0–50 kGy (0–5 Mrad) dla różnych intensywności dawek. Doświadczenia nad usuwaniem barwy ze ścieków włókienniczych były prowadzone przy użyciu sztucznych kąpiei zawierających barwniki, surfaktanty i inne związki chemiczne. Prowadzono również doświadczenia ze ściekami nasyconymi czystym tlenem. W napromieniowanych próbkach ścieków oznaczono barwę, pH, ChZT, BZT₅ i zawartość detergentów.

STRAHLENDISSOZIATIVER ABBAU VON FARBSTOFFEN IN WÄSSRIGEN LÖSUNGEN UND IN ABWÄSSERN DER TEXTILINDUSTRIE

Im vorliegenden Bericht werden Versuche zum strahlendissoziativen Abbau einiger Farbstoffe in wässrigen Lösungen und in Abwässern der Textilindustrie erörtert. Die Farbstoffe gehörten zu vier verschiedenen Handelsgruppen.

Die Lösungen wurden mit γ -Strahlen aus Co⁶⁰ in einer Radiationskammer bestrahlt. Die Kapazität der Radiationsquelle betrug 20 000 Ci. Die Kinetik der Entfärbung wurde mit verschiedener Intensität, die von 0–5 Mrad schwankte geführt. Für Entfärbungsversuche wurden synthetische Bäder hergestellt; sie enthielten Farbstoffe, Detergentien und andere Chemikalien. Untersucht wurden auch mit Sauerstoff gesättigte Abwässer. Analysiert wurden: Färbung, pH-Wert, CSB, BSB₅ und Detergentiengehalt.

РАДИОЛИТИЧЕСКОЕ РАСПРЕДЕЛЕНИЕ КРАСИТЕЛЕЙ В ВОДНЫХ РАСТВОРАХ И СТОЧНЫХ ВОДАХ ОТ ЗАВОДОВ ТЕКСТИЛЬНОЙ ПРОМЫШЛЕННОСТИ

В работах представлены попытки радиолитического распределения некоторых красителей в водных растворах и сточных водах от заводов текстильной промышленности. Испытания проводились на нескольких избранных красителях, представляющих 4 торговые группы.

Водные растворы облучались в радиационной камере лучами γ из Co⁶⁰. Полная активность источника излучения составляла 20 000 Ci. Кинетика обесцвечивания красителей в водном растворе исследовалась в пределах 0–50 kGy (0–5 Mrad) для различных интенсивностей доз. Испытания по удалению окраски из текстильных сточных вод проводились при использовании искусственных ванн, содержащих красители, поверхностно-активные вещества и другие химические соединения. Проводились также испытания по сточным водам, насыщенным чистым кислородом. В облученных пробах сточных вод определены окраска, pH, ХПК, БПК₅ и содержание detergentов.