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BY-PRODUCTS FROM THE ESSENTIAL OIL INDUSTRY - VALORIZATION AND RECYCLING. PRACTICAL APPLICATION - ADSORPTION OF TEXTILE INDUSTRY DYES

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Abstract

The purpose of this study is to enrich the approaches and methods for valorization and utilization of the essential oil industry by-products and to outline the perspectives in this area. As a possible practical application lavender and rose by-products were used as a bio adsorbents for removal of textile industry dyes. The influence of contact time, adsorbent amount and temperature was investigated. The efficiency of adsorption with lavender and rose by-products was compared with activated carbon, Al_2O_3 *and silica gel. The results suggested that lavender and rose solid by-products could be successfully utilized as bio sorbents for purification of textile industry waste waters.*

Key words: *adsorption, by-products, lavender, rose, textile dyes.*

INTRODUCTION

The essential oil and, in particular, the rose and lavender essential oil industry, is one of the emblematic and widely represented among the agricultural industries in Bulgaria. Essential oil and medicinal raw materials from various families (*Rosaceae*, *Lamiaceae*, *Asteraceae*, etc.) are grown and processed. Bulgaria, together with Turkey, provide over 80% of the world's supply of rose oil, with the Bulgarian one having better indicators and, accordingly, the most sought after by the perfumery and cosmetics industry. Although there are over 200 species of roses, the following cultivated roses are mainly used to obtain aromatic products: *Rosa damascena* Mill., *Rosa gallica* L., *Rosa centifolia* L. and *Rosa alba* L. (Kovacheva et al., 2010). Due to the low content of essential oil in the raw material (for example, for *Rosa damascena* about 0.030-0.045%; for *Rosa centifolia* and *Rosa alba*, these amounts are even lower: 0.02% and 0.015-0.030%), after its extraction (through steam distillation, extraction with organic solvents or liquefied gases: freons, CO_2) large amounts of by-products are generated. In most cases, distilleries dump this biomass into the surrounding area, where it spontaneously ferments. However, these procedures disturb the ecological balance in the areas where these wastes are disposed of, both because of water pollution and because of the antibacterial action of biologically active substances (polyphenols, residual aromatic components) in the by-products. Moreover, this approach leads to the loss of valuable substances with potential applications in medicine, food, cosmetics, etc. industries. In order to find solutions for the utilization of waste raw materials from the essential oil industry, various approaches and methods have been developed in recent years - controlled composting or co-composting with biodegradable waste materials, extraction of valuable biologically active substances such as polyphenols, polysaccharides, etc., biosorption of various industrial pollutants - heavy metals, pigments, etc., gasification, feed additives, etc. (Schieber et al., 2005; Slavov et al., 2016; 2017). In most cases, these approaches can be integrated into combined methods that provide more complete utilization of waste raw materials (Slavov et al., 2017).

Textile industry, although worldwide developed, with incomes above trillion dollars, and more than 35 million employees, is among the manufactures which uses large amounts of water and causing serious environmental problems (Lellis et al., 2019). One of the main concerns for the environment is related to safely disposal of the wastewaters in the open water basins. Besides the substances used for bleaching, textile pretreatment, levelling and finishing agents, regulators of the pH, etc., the dyes are among the compounds causing mostly environmental pollution (Nicolai et al., 2021). Having usually phenolic character they are stable to light, temperature and biodegradation which makes them resistant and difficult to degrade. Furthermore, some of the products of degradation, also could pose a serious health risks for the humans and the environment (Crettaz et al., 2020). Globally, around 10,000 dyes are commercially used and their overall production is around 700,000 tons annually (Singh et al., 2017). Often, the particular companies have their own receipt and usually more than one dye is included in the formulations. Direct discharge is related to increased biochemical oxygen demand and chemical oxygen demand (COD), and often leads to heavy pollutions (Dehghani et al., 2018). Therefore, effective methods to remove the dyes from wastewaters are needed. The approaches used for purification of the wastewaters comprises of coagulation, flocculation, precipitation, membrane filtration, electrochemical techniques, bioremediation by microorganisms and conventional biological treatments (activated sludge) (Amalina et al., 2022; Lellis et al., 2019). However, these techniques in many cases lack efficiency or necessitate high initial investments and expensive operative costs (Kumar et al., 2011).

The removal of pollutants from industrial wastewater streams is a major problem for industry, agriculture and society. Increasing water pollution is suggested by studies on water treatment, with the primary importance being the removal of heavy metals and organic pollutants from industrial wastewater and increasing its potability (Abdel-Ghani & Elchaghaby, 2007; Karthik et al., 2014). The adverse effects of heavy metals, even in small amounts, are diverse and include destruction of the immune system, interference with the synthesis of some vital enzymes, cancer and nervous disorder, especially in children (Karthik et al., 2014). Natural materials that are available in large quantities or some wastes from agricultural activities can potentially be used as low-cost adsorbents, as they represent resources that are renewable, widely available, and environmentally friendly. A large number of studies have focused on the application of low-cost natural adsorbents, including carbonaceous materials, agricultural wastes and by-products, which are recognized as a potential alternative to conventional technologies such as sedimentation, ion exchange, solvent extraction and membrane technologies for the removal of heavy metals and organic pollutants from industrial wastewaters, as these processes have technical and/or economic limitations. The plant biomasses had been found effective in removing trace metals and organic pollutants from the environment (Abdel-Ghani & Elchaghaby, 2007).

Adsorption is a process of pollutant removal that is among the most promising and effective techniques for wastewaters purification (Wong et al., 2020). The adsorbents could be often regenerated and reused (Nascimento et al., 2014). Besides, additional benefit is utilization of by-products from the food and agricultural industry (essential oil industry for example) as natural, biodegradable, cheap and readily available adsorbents (Slavov et al., 2017).

Available data on the use of rose and lavender industrial by-products for adsorption of pollutants (heavy metals, such as copper, chromium, oil, zinc, textile organic materials, etc.) indicate that these materials can be used for wastewater treatment (Abdel-Ghani & Elchaghaby, 2007; Bhatti et al., 2011; Iqbal et al., 2013). Rabbani et al. (2016) investigated applicability of rose watering waste (the residues after industrial water-steam distillation for essential oil production). They found that the rose by-products could be successfully used as adsorbent and optimum conditions toward maximum removing value of COD and the color were contact time of 60 and 45 min for bulk and nano-biosorbent, respectively, as well as pH = 5.0 and biosorbent dosage of 2 g/L.

Therefore, the aim of the present study was focused on utilization of by-products from the essential oil industry (rose and lavender solid residues) as natural, renewable and biodegradable adsorbents for removal of industrial textile spent dyes from waste-streams. This approach also allows searching for alternative methods for valorization of the industrial by-products of the rose and lavender essential oil industry.

MATERIALS AND METHODS

Lavender (bio-certified *Lavandula angustifolia* Mill., Sevtopolis var.) - L and rose (bio-certified *Rosa damascena* Mill.) - RD, by-products from industrial steam distillation of fresh plant material were provided by the ECOMAAT distillery (Mirkovo, region of Sofia, Bulgaria; crop 2021).

The textile dyes (initial and spent ones) were provided (as a ready to be used solutions) by E.Miroglio EAD - Sliven, Bulgaria.

The rose and lavender by-products were collected from the still after the end of industrial treatment and dried. The dried solid residues were washed with distilled water (100 g with 4×400 mL deionized water). The residual solid mass was dried, milled and sieved. The same procedure was followed for RD and L by-products washing with acetone, 70% ethanol and 0.1 N hydrochloric acid. For experiments RD and L fractions with particle size ranging from 50 µm to100 µm were used.

The dyes' adsorption was performed as follow: 20 mL dye solution was added to 1 g of RD or L residue in a 50 mL centrifuge tube and the tubes were placed on a laboratory shaker MLW THYS 2 (VEB MLW Labortechnik Ilmenau, Germany). The shaker was started (100 rpm) and at a specified time (2.5, 5, 10, 20, 30, 40, 60, 90 and 120 min) a centrifuged tube was removed from the shaker and the content was filtered through a paper filter and further through a syringe filter CA 0.45 µm (Isolab, Germany). Adsorption of the filtrate was measured in a 1 cm cuvette at 489 nm for dye 2, 578 nm for dye 3, 600 nm for dye 4 and 546 nm for dye 6, employing LLG-uniSPEC 2 UV-Vis spectrophotometer (LLG Labware, Germany). The concentration of the remaining after the adsorption dyes was calculated using a calibration curves, prepared with solutions of the dyes with known initial concentrations and proper subsequent dilutions.

The analyses were run in triplicate, and the data were given as mean values. Statistical significance was detected by analysis of variance (ANOVA, Tukey's test; value of $p<0.05$ indicated statistical difference).

RESULTS AND DISCUSSIONS

Removal of four type of dyes were studied in the present investigation: two reactive type dyes, one dispersed for polyesters and one metal-complex. In order to check the applicability of RD and L by-products as adsorbents, the dye № 2 (dispersed for polyesters) was firstly used before dyeing process (solution ready for dyeing provided from the manufacturer) and comparison was made with the same dye but used in the dyeing process (spent dye). The characteristics of the investigated dye mixtures are presented in Table 1.

Table 1. Characteristics of the textile dyes, objects of the investigations

	Dye	C, mg/ml	pH	pH spent dye
2	Dispersed - polyester (Dianix Flavin XF; Dianix Scarlet XF; Dianix Marine XF)	3.21	4.14	4.25
3	Reactive - cotton (Levafix Gelb CA; Levafix Rot CA; Levafix Marine CA)	2.57	6.09	10.79
4	Reactive - wool (Kemazolan Giallo W-CE/01; Lanasol Red CE; Lamasol Navy CE)	4.31	4.63	4.77
6	Metal-complex (Kemsetl Giallo 2R; Kemaset Ross G; Kemaset Marine Blue R/02)	4.77	5.80	5.12

Influence of the contact time on the adsorption of textile dyes

One of the most important parameter considering the adsorption as a physical process is the time when the adsorbent and the pollutant interact between each other. For this reason first the contact time of the adsorbent with the dyes was investigated. The contact time determines the equilibrium of the dye adsorption and is important from both economic and ecological matters (Figure 1). In the beginning of the adsorption the process takes place quickly because of the great number of unoccupied sites at the surface of the L and RD by-products. Sharp increase of the adsorbed azo dye was observed approximately until the 5th min for all the samples, after that clearly equilibrium was established.

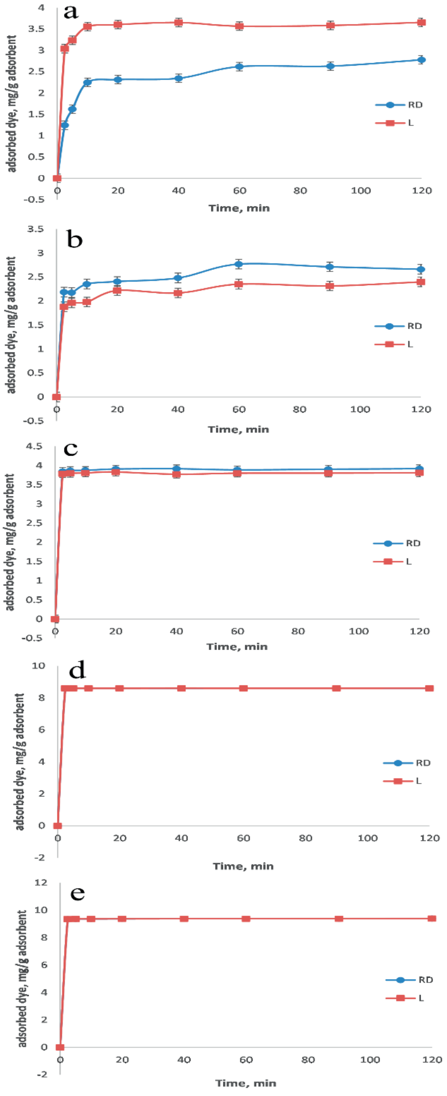

Figure 1. Influence of the contact time on the adsorption of textile dyes (amount of the adsorbent: 1 g; temperature: 20ºC; shaking at 100 rpm); a) initial dye 2; b) spent dye 2; c) spent dye 3; d) spent dye 4; e) spent dye 6

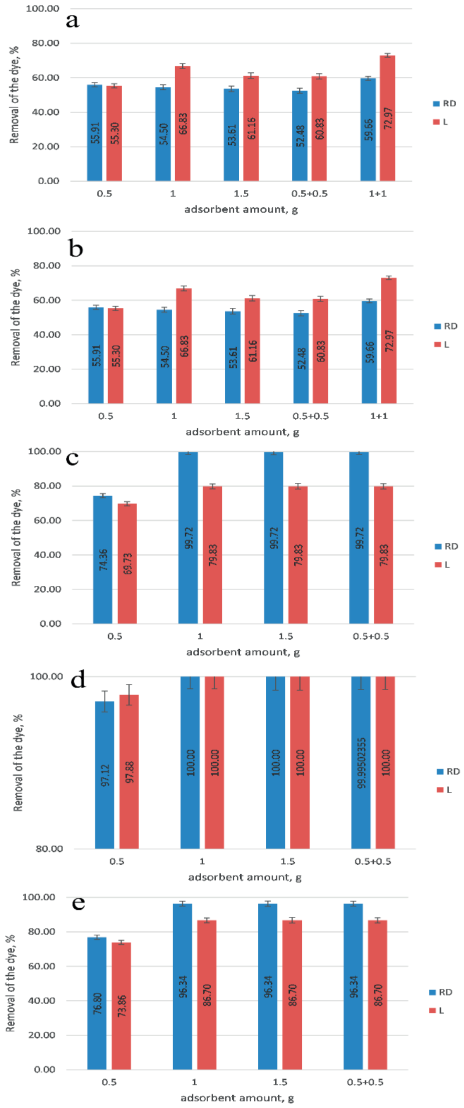

Figure 2. Influence of the amount of the adsorbent/re-adsorption on the adsorption of textile dyes (contact time 20 min; temperature: 20ºC; shaking at 100 rpm); a) initial dye 2; b) spent dye 2; c) spent dye 3; d) spent dye 4; e) spent dye 6

After 120 min when the process was stopped the efficiency of the adsorption was: for initial dye 2 - 58% for RD and 66% for L; for spent dye 2 - 60% for RD and 68% for L; for spent dye 3 - almost 100% for RD and 81% for L; for spent dye 4 - 100% for both adsorbents; for spent dye 6 - 94% for RD and 83% for L.

Similar results were obtained by Rabbani et al. (2016) investigating adsorption capacity of nanoparticles obtained from rose watering waste (*Rosa Damascena*). Clearly, the adsorption efficiency depended from the type of the dye mixture and for dye 2 L had the better results, while for dyes 3 and 6 - RD.

Influence of the amount of the adsorbent/re-adsorption on the adsorption of textile dyes

The amount of adsorbent plays important role and usually higher amounts lead to better effectiveness in the percentage removal of the pollutant. This, of course is related also to the higher operating costs and when possible lower amounts of the adsorbing material should be employed. Besides, the spent adsorbent have to be further treated in order to be safely disposed. The best results were achieved (Figure 2) using 1 or 1.5 g RD adsorbent (single adsorption; almost 99% removal for spent dye mixtures 3, 4 and 6 was achieved). The maximum possible single amount of adsorbent was 1.5 g - above this amount retention of the water phase inside the adsorbent did not allowed to obtain purified water solutions.

Consecutive adsorption was also tested and it was found that for both adsorbents doses of 0.5+0.5 g and 1+1 g adsorbents showed better results only for dye 2. One of the problems arising using consecutive adsorption was related to the higher water retention capacity of the R and L, having strong hydrophilic character. At doses 1.5+1.5 g adsorbents no water phase was able to obtain after adsorption. In our experiments single adsorption procedure with 1 g or 1.5 g of both adsorbents showed comparable results with consecutive adsorption, suggesting that one step was enough. For dye 2, however, the results were not the best ones, and additional steps or other adsorbents should be employed.

Influence of the temperature on the adsorption of textile dyes

The results for the effect of temperature on the effectiveness of adsorption for both adsorbents was ambiguous for different dye mixtures. For dye number 2 was even different for the spent and initial dye mixtures. For the initial dye the best removal was observed at 40°C. At 100°C the removal was around 80% but at this elevated temperatures it is possible to have degradation of some of the dyes which would gave misleading results. For the spent dye 2 the adsorption was negatively influenced by the temperature increase (Figure 3). For the dye 3 again a negative trend with temperature increase was observed. Dye mixtures 4 and 6 were not influenced by the temperature and this is the best possible situation, having in mind that operating at lower possible temperatures (ideally at ambient temperature) is beneficial for energy saving.

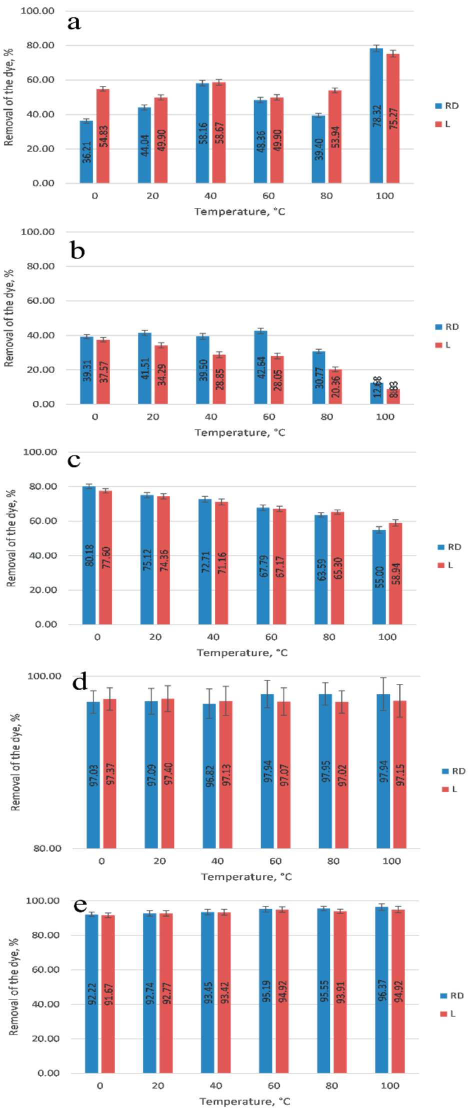

Figure 3. Influence of the temperature on the adsorption of the textile dyes (contact time 20 min; amount of the adsorbent: 1 g; shaking at 100 rpm)

Tosi Pelosi et al. (2013) observed that with temperature increase the adsorption of acid orange 7 dye by *Salvinia natans* biomass was favored. Balarak et al. (2020) had similar conclusions on the influence of the temperature on the adsorption of acid orange 7 by canola wastes. However, Wu et al. (2018) observed ambiguous effect of the temperature on the adsorption of acid orange 7 on waste brewery's yeast. The results obtained by the authors might be due to the increased rate of desorption with temperature increase. All these observations suggested that the temperature influences the adsorption process most of the time depending from the adsorbent, pollutant and the matrix. No significant effect (favoring or suppressing) for the influence of the temperature on some of the particular adsorbent (RD or L) used was observed in the present study.

Influence of the type of the adsorbent on the adsorption of the textile dyes mixtures

In the next experiments the effectiveness of the RD and L to remove dyes from wastewaters was compared with several known commercial adsorbents: activated carbon, Al_2O_3 and silica gel (Figure 4). Also comparison of the adsorption applicability with pretreated RD and L by-products (with 70% ethanol, acetone and 0.1 N hydrochloric acid) was made. The best results for adsorption effectiveness of the initial dye 2 showed the silica gel but for the adsorption of the mixture of spent dye 2 the acid washed lavender by-product (L_Res_AE) was the most effective followed by Al_2O_3. RD had better adsorption capacity than L but in general their adsorption effectiveness towards dye number 2 was limited. Spent dye 3 was well absorbed by most of the adsorbent used and again L_Res_AE was the most effective. For the spent dyes 4 and 6 activated carbon, silica gel and L_Res_AE showed the highest effectiveness. The results suggest that acid washing is generally beneficial to the applicability of the residues as adsorbents. These observations are in accordance with the results for the influence of the pH on the adsorption of Acid Orange 7 and Remazol Black 5 reactive dyes from aqueous solutions (Hamzeh et al., 2012). The effectiveness at lower pH is related to the net charge of the adsorbents. At lower pH medium the functional groups in the plant matrix will be protonated and attraction forces will favor retention of the dyes.

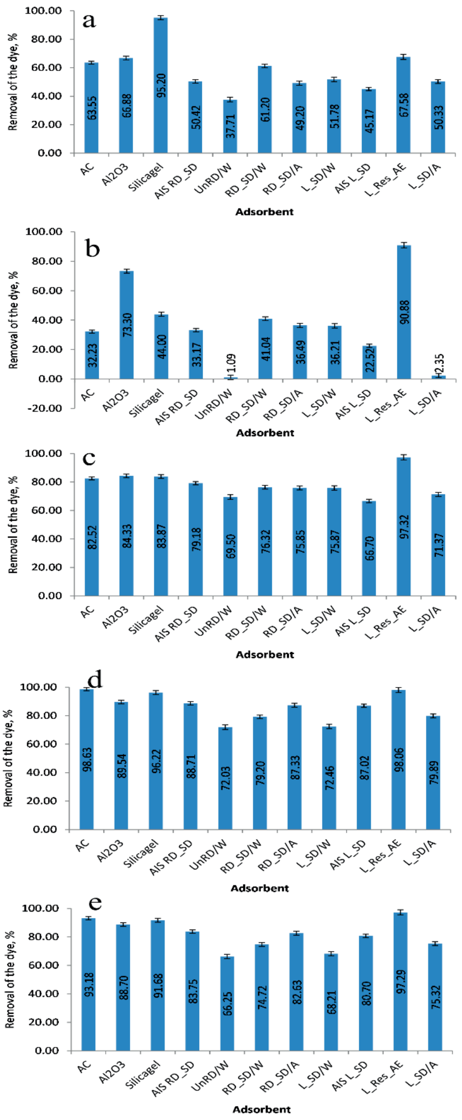

Figure 4. Influence of the type of the adsorbent on the adsorption of textile dyes (contact time 20 min; amount of the adsorbents: 1 g; shaking at 100 rpm). AC - active carbon; Al_2O_3; Silicagel; AIS RD_SD - rose by products washed with 70% ethanol; UnRD/W - dried rose petals (non-distilled) washed with distilled water; RD - steam-distilled rose by-products washed with distilled water; RD_SD/A - rose by products washed with acetone; L - steam-distilled lavender by-products washed with distilled water; AIS L_SD - lavender by-products washed with 70% ethanol; L_Res_AE - steam-distilled lavender by-products washed with 0.1 N HCl; L_SD/A - lavender by products washed with acetone

The results from the experiments suggested that RD and L by-products could be successfully applied as a bioadsorbent for removal of textile dyes from industrial water solutions. The residues generated after adsorption of the dyes could be used as a substrate for further higher fungi degradation and production of mycelium based biocomposites (Angelova et al., 2021). Due to the phenolic nature of the most of the dyes, the higher fungi, able to produce lignocellulosic enzyme complexes, could successfully degrade the organic pollutants and completely eliminate the hazardous threat for the nature these dyes pose.

CONCLUSIONS

The present study explored the application of two industrially generated by-products of the essential oil rose and lavender industry as bioadsorbents. The solid by-products are cheap, renewable, biodegradable and natural materials, which could be successfully applied for adsorption of organic pollutants, such as the spent dyes in the industrially generated textile wastewaters. The influence of contact time, amount of the adsorbent, temperature, and initial pretreatment of the adsorbents was investigated. Comparison of the effectiveness of the RD and L as adsorbents with activated carbon, Al_2O_3, and silica gel was made. It was found that equilibrium was established after 5-10 min contact time. The influence of the temperature on the dyes' removal was ambiguous and for this reason the ambient temperature was chosen for adsorption experiments (20°C). Previous studies of our team on the adsorbents particle size influence suggested that particles around or less than 50 μм showed the best results for adsorption effectiveness. Acid washing (with 0.1 N hydrochloric acid) as a pretreatment lead to the best results for removal of textile dyes from the medium. The resulted adsorbents had adsorption capacity similar to that of activated carbon and silica gel as a well-known commercially available adsorbents. The best adsorption effectiveness showed both acid-washed rose and lavender adsorbents, as lavender acid washed by-product adsorbed almost all of the dyes in spent formulations 3, 4 and 6.

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