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Layered compact textiles applied in fixed-bed column as filters for dye-rich textile wastewaters treatment—a case study

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ABSTRACT

The presented case study examined the possibility of using four commercially available compact textiles in small-scale column purifying system conducted in a continuous-flow operation as filters for colour, organic pollutants and salt reduction. Two wastewaters were prepared for the experimental purposes by combining chemically different dyestuffs, auxiliaries and chemicals in order to simulate dye-bath effluents from cotton (reactive) and wool (acid) dyeing, respectively. Treatment of the laboratory-prepared wastewaters was carried out in columns packed with alternating layers of sand and individual compact textile and, for comparison, in a column comprised merely of sand. It was found that both non-woven textiles, either made from polypropylene (PP) or bicomponent PP/polyethylene (PE) yarn with more complex structure and higher total void area, were more suitable for adsorption/filtration of colour and organic pollutants from dye-rich textile wastewaters in comparison to both woven fabrics. Monitored pollution parameters in the initial and outflow samples indicate that the sand/non-woven system reduced colour by up to 71%, and also appreciably lowered the organic pollutants in both dye-rich wastewaters depending on the wastewater's composition and trial duration. The efficacy of the control columns was attained—maximally 30% of colour and total organic carbon reduction. Generally, the system showed an explicit buffering capacity, and on the other hand, negligible reduction of the salt content.

Keywords: Compact textiles; Textile wastewaters; Biofilter; Decoloration; TOC reduction

1. Introduction

Fibre-forming polymer materials used in wastewater treatment systems, e.g. filtration media, or for environmental protection, e.g. erosion protection and the sealing of toxic waste, are principally divided into four groups: (geo)textiles, (geo)membranes, (geo)nets, (geo)composites and mixtures of these groups [1].

The three most commonly used fibre-forming polymers in hazardous waste facilities are polypropylene—PP, polyamide—PA and polyethylene—PE, but polyester (polyethylene terephthalate [PET] or polybutylene terephthalate [PBT]) is almost inevitably used when high-strengths are required [2]. There are other

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special higher-strength polymers available on the market but some of them are unavailable in large quantities or tend to be very expensive. Textiles made from such kinds of polymers are generally non-woven, heat-bonded or needle-punched and woven textiles, basically designed from three types of yarns, namely monofilament, multifilament and staple-fibre yarns.

Generally, the compact textile materials themselves or in combination with natural materials (sand, gravel, zeolite, etc.) are used for filtration or separation and, in a few cases, for wastewaters treatment in bioreactors/biofilters with fixed-beds [1,3,4]. The key element for the design and efficient operation of a textile-based system is the selection of an appropriate textile material, which should be characterized by its availability, low-cost, physical-mechanical properties (breaking strength, flexibility, surface charge, etc.), excellent environmental compatibility, biological and chemical stabilities and sufficient permeability or hydraulic conductivities (also after long-term operations) [2]. A high-texture surface area must be available for active biomass growth, with pore sizes large enough for undisturbed wastewater flow through the system.

The pollutants' removal mechanism in biofilters using textiles as packing material seem to be as follows: surface and/or internal filtration of suspended organics, growth of an active biomass, absorption of dissolved pollutants by the attached biofilm and finally, biodegradation [5].

In the earlier stages of wastewater treatment, when the biofilm is thin, larger insoluble pollutants' particles may be filtered and absorbed on the surface of the packed materials. The filtration performances of the textiles are governed by several factors. The most important is permeability, which can vary immensely depending on the construction of the fabric. The filtration functions of textiles can fail by virtue of micro-organisms multiplying and blocking the pores, or by chemical precipitation from mineral substances blocking the pores.

After long-term functioning and suitable conditions, micro-organisms could grow on the surface producing a corresponding thickening of the biofilm required for the biological conversion of pollutants in less harmful products (CO_2, H_2O, different inorganic substances, etc.) [6]. Biomass growth reduces pores size, the surface area available for mass transfer of either wastewater or oxygen into the biofilm declines, and can thus eventually clog the system and diminish treatment efficiency after a lengthy operation (Fig. 1).

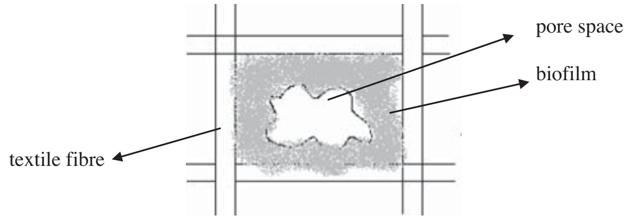


Fig. 1. Model of attached biomass growth in textile fibres' pores [8].

Biomass structure and growth is a function of different physical, biological and chemical factors, such as the type and texture of a compact fibre-forming material, the flow, type and concentration of the pollutants, mass transfer, temperature, pH, oxygen content, microbial population type, the physiology of the cells, the presence of nitrogen- and phosphorus-containing organic compounds, etc. [7].

Therefore, the purpose of this research was to focus attention on the adsorption and (bio)filtration abilities of various woven and non-woven textiles, packed in a column together with sand as a support media, for the reduction of colour, organic pollutants, pH and salt from differentially composed dye-rich textile wastewaters, regarding the structure and physical–mechanical properties of the selected textiles, as well as the chemical structure of the dyestuffs and the composition of the wastewater.

2. Materials and methods

2.1. Compact textiles

Four types of commercially available compact textiles with varying physical–mechanical properties were used during column trials. Two woven fabrics were made from PA and polyester (PES) fibres and two non-woven fabrics according to the heat-bonding procedure:

(1) From 100% PP yarn (30% Trevos and 70% Danaklon) with a length of 40 mm and fineness of 2.2 dtex.
(2) From bicomponent PP/PE yarn (Danaklon ES-C), where the core was from PP coated with PE, with a length of 40 mm and fineness of 2.2 dtex for applications requiring higher-strength.

2.2. Composition of the synthetically prepared dye-rich wastewaters

The experiments were accomplished using two synthetically prepared wastewaters differing in applied dyestuffs, auxiliaries and chemicals, in order to establish the (bio)filtration efficiency of the presented case study's column experiments for the treatment of diverse dye-bath effluents.

The initial "alkaline" dye-bath's wastewater characteristics for cotton dye-house effluents, contained 0.03 g/L of reactive vinylsulphone dye C.I. Reactive Black 5 (RB5), 0.3 g/L of a sequestering agent Alvirol AGK (Textilcolor), 0.3 g/L of an anionic wetting and de-aerating agent Cibaflow PAD (Ciba), 2 g/L of NaCl and NaOH for regulating the wastewater's salinity and alkalinity (pH 9–10), respectively. On the other hand, the initial "acidic" wastewater was composed as a typical dye-bath effluent from wool dyeing containing 0.03 g/L of acid dye C.I. Acid Orange 33 (AO33), 0.5 mL/L of an amfoteric levelling agent Keriolan A2 N (Bezema), 0.1 mL/L of pH-regulator Meropan EF (Bezema), 0.2 g/L of $(NH_4)_2SO_4$ and CH_3COOH (80%) for the regulation of the wastewater's acidity (pH 5–6), respectively. The chemical structures of selected dyestuffs and their colour indexes are presented in Fig. 2. All chemicals employed during the trials were of analytical grade: sodium hydroxide (NaOH), acetic acid (CH_3COOH) and ammonium sulphate ($(NH_4)_2SO_4$) were purchased from Fluka, and sodium chloride (NaCl) from Merck.

2.3. Laboratory-scale treatment assay

A continuously fed laboratory-scale experimental set-up was designed from a plexiglass column with a diameter of 6 cm and a length of 36 cm, with inlet and outlet pipes, a plastic reservoir for wastewater storage equipped with a stirrer for constant mixing, and a pump for attaining a continuous wastewater flow through the filled column of approximately 30 mL/h, and consecutively, a retention time between 12 and 14 h (Fig. 3). Columns, which provide an empty volume of 1L were fortified with a PP net and packed with:

(1) Washed sand (control column) with particle size of 2–12 mm, with a bed height of 28.5 cm, packed weight of 625 g and bed-porosity of 0.36 and
(2) An alternating strata of washed sand (2–12 mm) and one layer of individual compact textile, with a bed height of 28.5 cm (as in the column with sand), in order to establish the potential of two woven PA and PES, and two non-woven PP and bicomponent PP/PE (BICO) textiles to behave as filters for colour, salt and organic pollutants reduction from two synthetically prepared dye-rich textile wastewaters.

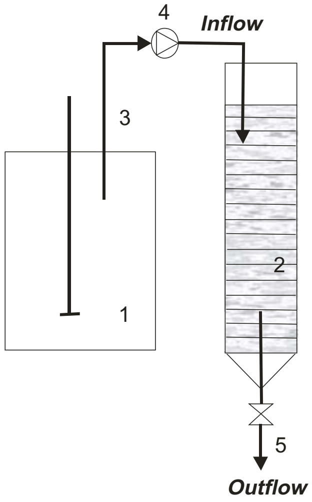
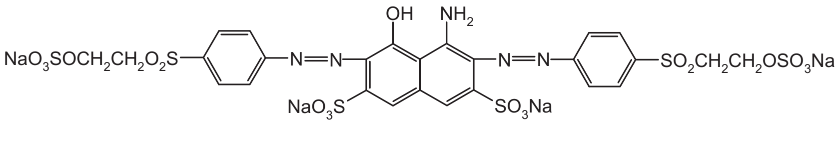
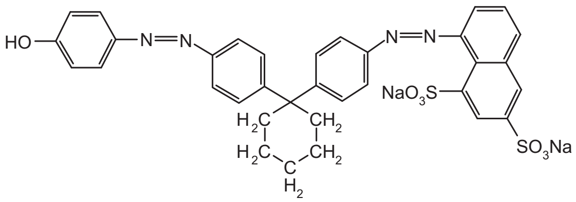


Fig. 3. Experimental set-up of wastewater treatment: 1—reservoir for wastewater, 2—fixed-bed column, 3—inlet pipe, 4—pump and 5—outlet pipe with a valve.



C.I. Reactive Black 5 (RB5)



C.I. Acid Orange 33 (AO33)

Fig. 2. Chemical structures and colour index (C.I.) generic names of two selected dyestuffs.

The sand in the column served as a filtration barrier for larger pollutants' particles and as a support material for thin textile layers. Particle size plays an important role in relation to system efficiency; large-sized sand particles ensure quick infiltration and thus preventing water retention and algal growth, small-sized particles influence higher treatment ability, but on the other hand, with greater clogging possibilities.

The hydrodynamic conditions in the inflow were continually controlled, and the volume of wastewater at the outflow was simultaneously measured. All experiments were performed at an ambient temperature of 22 ± 2˚C.

2.4. Analytical methodology

First, some mechanical properties such as the tensile strength, elongation at break and the breaking tenacity of the used textiles were measured according to standard DIN 53857, using a Textechno statigraph *M* test machine. In addition, certain other physical–mechanical properties were determined, such as thickness, weight and density, according to standardized methods. Field parameters, i.e. the total void area, total void area percentage, total void parameter and the number of voids per mm^2, of the four selected textiles, which affect the wastewater flow or even clogging, were measured by means of an Axiotech 25 HD (+pol) microscope (zeiss), equipped with an AxioCam MRc (D) high-resolution camera and KS 300 Re. 3.0 image-analysis software. The cover factor (CF) in percentage was calculated according to Eq. (1).

$$\mathrm{CF}(\%) = 100(\%) - \text{total void area}(\%) \tag{1}$$

A total of 20 measurements were taken for each sample. The field factor's measuring procedure has been fully described elsewhere [9].

Secondly, an assessment of the column experiments' efficiency was verified by monitoring several pollution parameters, i.e. absorbance, total organic carbon (TOC), pH as well as electrical conductivity (EC) and oxidation–reduction (redox) potential (ORP) in the initial and treated wastewaters. Samples for measurement were taken from the outflow every day with the exception of Saturdays and Sundays, and directly analysed (only the selected results are presented here). For qualitative information about colour changes, the absorbance was monitored at the wavelength of an individual dye's absorption maximum, for RB5 at 600 nm and for AO33 at 488 nm, using a Carry 50 UV-vis spectrophotometer (Varian) by means of a 10 mm quartz cuvette, according to standard EN ISO 105-Z10. The spectral absorption coefficient was calculated at wavelengths of 436 nm, 525 nm and 620 nm, according to Eq. (2).

$$\mathrm{SAC}(\mathrm{m}^{-1}) = \frac{A}{d} \times f \tag{2}$$

where A is the measured absorbance at a defined wavelength; d is the cuvette's diameter (mm); f is the factor for the conversion (f is 1,000).

Prior to absorbance measurement, the effluents were centrifuged for 10 min at 3,000 rpm, in order to prevent turbidity. The pH of the synthetic wastewaters was measured in regard to the ISO 10523 standard, using a MA 235 pH/ion Analyzer (Mettler Toledo). The TOC was measured by means of a DC-190 Analyzer (Dohrmann), in accordance with the ISO 8245 standard. The electrical conductivity was determined using MultilabP5 (WTW) and the redox potential was monitored by platinum electrode vs. the Ag/AgCl reference electrode filled with KCl electrolyte, connected to a MA 235 pH/ion Analyzer (Mettler Toledo).

Table 1
Properties of textiles used in the presented research

Properties:	PA woven	PES woven	PP non-woven	Bico PP/PE non-woven
Thickness (mm)	0.39	0.49	0.22	0.23
Mass (g/m^2)	111	160	20	22
Density (threads/10 cm)				
Warp	450	220	/	/
Weft	230	200		
Elongation at break (%)	76.7	49.9	40.9	9.7
Tensile strength (*N*)	975	1,261	43	60.3
Breaking tenacity (cN/tex)	487.7	630.7	132	77.2
Weave	Twill	Plain	/	/

3. Results and discussion

3.1. Analysis of compact textiles

Four commercially accessible fabrics were employed for the presented research; two woven and two non-woven textiles, made from the more commonly used fibre-forming polymers for hazardous waste facilities. Some physical–mechanical properties, as well as structural parameters, of all four fabrics were characterized and the results are depicted in Table 1.

Several field factors of the fabrics were measured by a microscopic method, in accordance with a predefined macro, thus ensuring that all samples were analysed in the same way and under the same conditions, as it was assumed that these parameters had the greatest influence on the ability of the fabrics to act as filters in fixed-bed systems for textile wastewater treatment. The obtained results are gathered in Table 2. At the same time, microscopic photos of all four compact textiles were taken, and are shown in Fig. 4.

From Fig. 4, it can be concluded that both dyestuffs, as well as the used auxiliaries, could be easily filtered and adsorbed on, preferably, PA and PES rough-structured and small-sized pores' surfaces. On the other hand, a higher total void area in percentages and a superior number of voids/mm^2 of non-woven textiles compared to woven, implied better wastewater infiltration and, consecutively, fewer problems with system clogging. From the results gained from absorbance, TOC and EC measurement (Figs. 5 and 6 and Table 3), it can also be perceived that the used non-woven textiles had enlarged treatment abilities; although adsorption of the negatively charged reactive dye RB5 can be hindered by the negatively charged sand and textiles [10]. After a prolonged period of system function, due to the low velocity-flow and long retention times, the well-formed fibre pores of the textiles could be grown with various micro-organisms' populations, presumably.

3.2. Treatment efficiency

Estimation of the column's performance packed with alternating layers of sand/compact textiles and for comparison solely with sand, was established by monitoring the pollution parameters, such as absorbance, pH, TOC and EC. The system was operated continuously without backwashing at a velocity-flow of approximately 30 mL/h and a retention time 12–14 h. These conditions were preliminarily determined, as the best trial conditions for maximal dye-rich wastewaters' decoloration and adsorption/filtration. The results obtained by UV–vis spectroscopic measurement during a treatment period of 40 days are gathered in Figs. 5 and 6, separately for "alkaline" RB5 dye-rich wastewaters and "acidic" AO33 dye-rich wastewaters.

Figs. 5 and 6 display the observed reductions in absorbances, measured in both the "alkaline" and "acidic" wastewaters at the wavelength of maximum absorption, respectively, over the 40-day trial period. During the first few days of the trials, the absorbances were exceedingly low, irrespective of wastewater composition, owing to the dye filtration/adsorption ability of the packed material, but thereafter, the absorbances increased and stabilized at a certain value (acclimatization period of 10–12 days) and after 15–20 days decreased, depending on the applied dye and type of used textile. Generally, the greatest overall decolouration was achieved using both the non-woven fabrics ahead of both the woven fabrics. On the last day of the treatment experiments, the percentage of reactive RB5 dye removal from the wastewaters was 52% (sand/BICO), 47% (sand/PP), 35% (sand/PES), 31% (sand/PA) and 21% (control column with sand) and the percentage of acid AO33 dye was 71% (sand/BICO), 60% (sand/PP), 47% (sand/PA), 30% (control column with sand) and 20% (sand/PES).

To better understand the role of compact textiles and their adsorption/filtration efficiency during wastewater treatment, wastewaters' absorbances were scanned throughout a UV/Vis spectrum, from a

Table 2
Field factors of used textiles (mean value of 20 measurements)

Sample	Total void area (%)	Total void area (mm^2)	Total void parameter (mm)	No. of voids/mm^2	CF[a] (%)
PA	0.08	0.03	2.83	11.17	99.92
PES	0.98	0.22	8.93	10.47	99.02
PP	15.74	2.29	189.33	440.43	84.26
Bico PP/PE	15.36	2.46	177.15	413.38	84.64

[a] CF—calculated values according to Eq. (1).

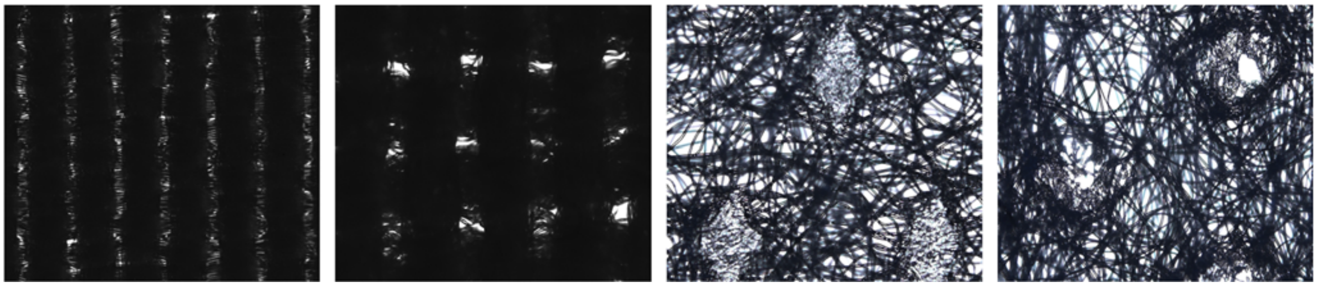


Fig. 4. Microscopic pictures of PA, PES, PP and bico PP/PE, enlarged 5 times.

wavelength of 200 nm up to 700 nm, in the initial and treated samples after the 40th day of the trial period. The obtained results are demonstrated separately in Figs. 7 and 8 for "alkaline" and "acidic" wastewaters.

From Fig. 7, an explicit curve could be observed, measured in the initial "alkaline" wastewaters, with a high absorbance maximum of 1.0395 at a wavelength of 604 nm and two peaks in the UV region, i.e. a major at a wavelength of 315 nm and a minor at 392 nm, which are characteristic for reactive RB5 dye. Absorbances were diminished throughout the entire UV/Vis spectrum in all columns on the last day of the presented experiments, presumably due to dye filtration and/or adsorption. Also, hipsochromic shift of the absorbance maximum occurred in those columns within the Vis region from 604 nm to 588 nm (columns filled with sand, sand/PA and sand/PES), and from 604 nm to 564 (columns filled with sand/PP and sand/BICO). Maximal absorbances within the UV region were also changed, implying a modification of the RB5 dye structure. Gradually, on account of the longer retention time, anaerobic or anoxic conditions arose in the biofilter's inferior layer, under which azo dyes readily reductive cleaved via four-electron reduction at azo linkage [11–13] and, consequently, the colour of the treated-samples perceivably changed from dark blue, to reddish-blue or to bright violet, respectively.

The absorption characteristics of the dyestuffs were modified during wastewater treatment [14] and consecutively, the absorbance of model's wastewaters decreased, whilst the visible colouring remained, as was also evident from the calculated SAC values (Table 3).

The same results as discussed above were noticed when trials with wastewater containing AO33 were

Table 3
The monitored parameters in initial and treated "alkaline" and "acidic" wastewaters

	TOC (mg/L)	EC (μS/cm)	pH	ORP (mV)	SAC at 436 nm (m^{-1})	SAC at 525 nm (m^{-1})	SAC at 620 nm (m^{-1})
Alkaline							
Initial	143	1,070	9.2	−60.8	32.83	50.12	88.03
Sand	100	859	7.9	42.6	28.48	41.07	69.69
Sand/PP	65	716	7.9	30.2	19.11	22.13	18.07
Sand/BICO	47	755	8.2	10.5	17.54	26.40	34.09
Sand/PA	92	915	7.6	22.1	26.26	32.88	29.37
Sand/PES	81	1,015	8.2	−24.5	22.34	39.82	59.48
Acidic							
Initial	260	657	6.3	77.5	27.28	34.23	0.68
Sand	218	634	8.0	106.5	20.73	23.68	1.99
Sand/PP	89	627	8.0	66.9	9.88	14.24	0.96
Sand/BICO	135	650	7.9	104.1	7.42	10.50	1.36
Sand/PA	156	624	8.2	55.9	14.62	17.74	1.35
Sand/PES	160	562	7.9	72.5	23.41	27.96	3.05

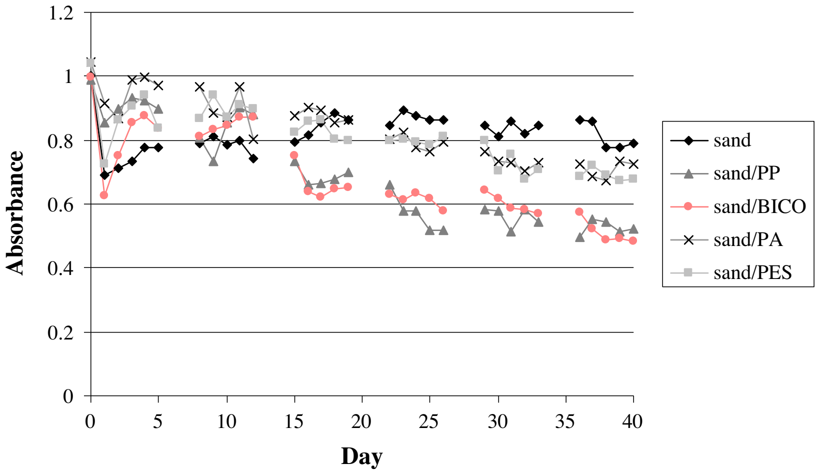


Fig. 5. Absorbance of initial and treated “alkaline” wastewater including RB5 dye.

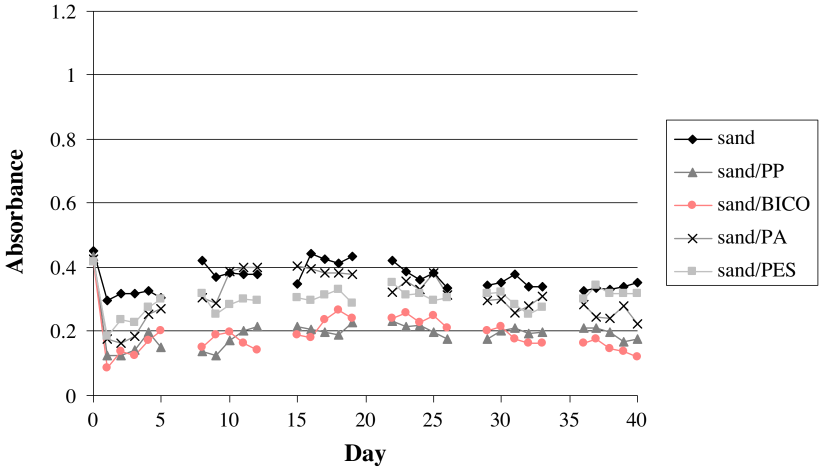


Fig. 6. Absorbance of initial and treated “acidic” wastewater including AO33 dye.

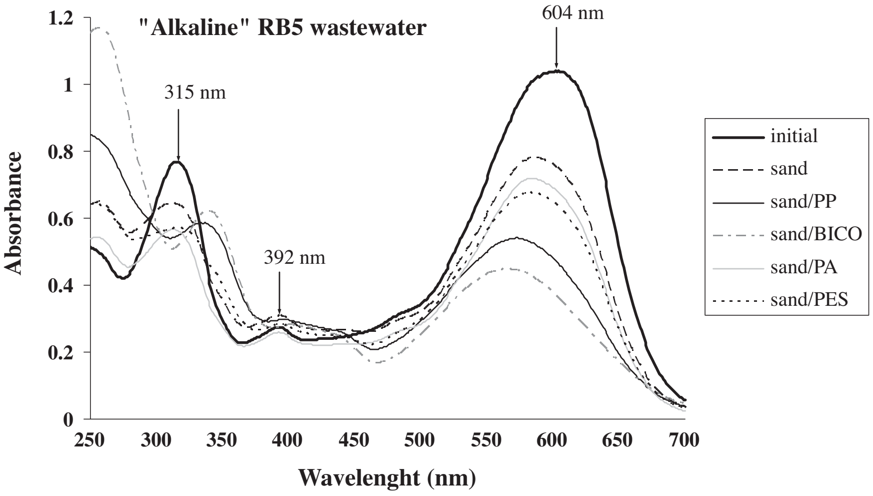


Fig. 7. UV/Vis spectra of initial and treated “alkaline” wastewaters including RB5 after 40th day.

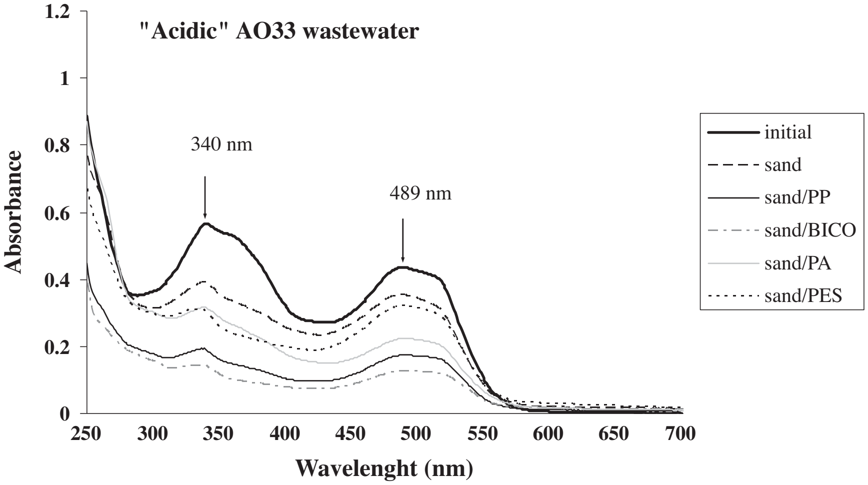


Fig. 8. UV/Vis spectra of initial and treated "acidic" wastewaters included AO33 after 40th day.

conducted (Fig. 8). During the 40th day experiment, a change in colour intensity was also manifested, although the wavelength of the absorbance maximum remained invariant at 489 nm and remained unchanged. Absorbance substantially decreased from 0.436 to 0.126, depending on the column's filled material.

Table 3 summarizes several monitored parameters: TOC, EC, pH and redox potential (ORP), as well as the calculated spectral absorbance coefficients at wavelengths of 436, 525 and 620 nm in both the initial and treated wastewaters after 40-day period, separately for "alkaline" and "acidic" dye-bath effluents.

It can be observed from Table 3 that the initial TOC values measured in both synthetically prepared wastewaters were very high, 143 and 260 mg/L, respectively; preferentially on account of auxiliaries' presence and, in the case of "acidic" wastewater, also due to the addition of acetic acid. The best organic pollutants removal on the last day of the experiment was detected in the "alkaline" dye-bath wastewater flowing through the column filled with a combination of sand and bicomponent PP/PE yarn, i.e. the reduction rate was from 143 to 47 mg/L (for 67%); followed by 66% TOC reduction in the "acidic" dye-bath wastewater treated by a combination of sand/PP (from 260 mg/L to 89 mg/L). The minimum TOC reduction was observed in those trials when only sand was employed in the column, i.e. from 260 to 218 mg/L (16%) in the "acidic" waste-bath and from 143 to 100 mg/L (30%) in the "alkaline" waste-bath.

The EC that depended on the salt content was importantly decreased only during the treatment of the "alkaline" wastewaters from reactive dyeing, which usually contain larger amounts of various salts taking an active part in the reduction of surface tension on the boundary layer during dyeing. Therein, the remaining salts in the wastewaters could improve dye uptake during treatment, as some investigations revealed a connection between dye adsorption and the applied electrolyte ionic species, depending on their natures and concentrations [15,16].

The initial pH values were significantly lowered at the outflow after 40 days in the case of the "alkaline" dye-bath wastewaters, and enlarged when the "acidic" wastewaters were treated, irrespective of the packed material. Evidently, the fixed-bed column showed an explicit buffering capacity as was already expected from reported researches [14,17].

The oxidation–reduction potential at the inflow was higher in the "acidic" wastewater, indicating higher oxygen content in comparison with the ORP measured in the initial "alkaline" wastewater. The ORP values changed during the experiment regarding the ORP of the filled materials, treatment and retention time, etc. Besides the above-listed factors, ORP depended greatly on several operational parameters such as external temperature, pH, reactions in wastewater, oxygen content, measuring time, etc., therefore, the reported results probably did not reflect the real image.

4. Conclusions

The presented case study combined compact fibre-forming polymers and sanitary engineering, investigating the feasibility of using previously woven and non-woven textiles in order to reduce selected parameters from two synthetically prepared dye-rich wastewaters, by physical filtration and adsorption. Specifically, the presented system reduced the colour by up to 71% and TOC by up to 67%, depending on the wastewaters' compositions, as well as column operational parameters such as, treatment time and, above all, the type of bedding material used. Nevertheless, the field parameters of the selected textiles and their surface textures played a crucial role in the dyes and TOC reduction, because they influenced the textiles' solid filtration, adsorption, ion exchange and presumably micro-organisms' growth ability. It was found that a superior treatment efficiency of dye-bath effluents was attained using the treatment system filled with alternating layers of sand and non-woven fabrics, either made from PP or bicomponent PP/PE yarns.

This concept appears to be particularly applicable for small volumes of textile wastewater, and also for tertiary treatment.

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