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Treatment of desizing wastewater by catalytic thermal treatment and coagulation

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ABSTRACT

In the present study, the coagulation of the fresh and thermally treated desizing wastewater has been reported. The maximum COD reduction of fresh desizing wastewater using coagulation was observed with commercial alum at initial pH 4. This was followed by aluminum potassium sulfate (pH 4), $FeCl_3$ (pH 6), PAC (pH 6) and $FeSO_4$ (pH 4). The maximum COD reduction observed at a coagulant (commercial alum) dose of 5 kg/m^3 and pH 4 was 58% whereas the color reduction at these conditions was 85%. The results reveal that the application of coagulation on the catalytic thermal treated effluent is more effective in removing nearly 88% of COD and 96% of color at above mentioned conditions except at a coagulant dose of 1 kg/m^3. The amount of inorganic sludge generated gets drastically reduced (almost 25%) due to the reduced amount of coagulant. The COD and color of the final effluent were found to be 98.6 mg/l and 2.67 PCU, respectively, and the COD/BOD_3 ratio was 1.36. The settling rate of the slurry was found to be strongly influenced by treatment pH. The slurry obtained after treatment at pH 12 settled faster in comparison to slurry obtained at pH 4. The filterability of the treated effluent is also strongly dependent on pH. pH 12 was adjudged to be the best in giving highest filtration rate.

1. Introduction

A typical textile processing consists of desizing, scouring, bleaching, mercerizing, dyeing operations. The effluent generated contains a wide range of contaminants, such as surfactants, scouring agents, oil and grease, and oxidizing and reducing agents. The major contributor to chemical oxygen demand (COD) is the effluent from desizing operation, with a typical COD of over 10,000 mg/l. This high COD is from the sizes applied to cotton or synthetics to aid the process of weaving. The typical sizes include starch, polyvinyl acetate, polyacrylate and carboxymethyl starch, etc. [1,2].

There are four general methods for wastewater treatment namely chemical oxidation, biochemical oxidation, incineration and wet air oxidation [3]. Thermochemical precipitation and coagulation may be used as pretreatment steps. Thermolysis is a chemical process, by which a substance is decomposed into other substances by use of heat [4–6]. The catalytic thermolysis of desizing wastewater at 95 °C and autogenous pressure, in the presence of $CuSO_4$ catalyst, has proved its efficiency in reducing the COD by about 71.6% and color by about 82.7%, with substantial energy recovery in terms of solid residue [7]. Surfactants and dyes with high molecular weights are successfully removed by coagulation/flocculation processes followed by sedimentation, flotation and filtration. The advantage of the coagulation process is decolorization of the waste stream by the removal of dye molecules from the effluent, and not due to any decomposition of dye molecules, which may lead to an even more potentially harmful and toxic aromatic compounds [8].

Kumar et al. [9] investigated catalytic thermal treatment (thermolysis) of desizing wastewater for the reduction of COD and color under moderate temperature and atmospheric pressure conditions using various catalysts. In present study, treatment of fresh desizing wastewater and catalytic thermally treated desizing wastewater was carried out using different coagulants, such as, commercial alum, aluminum potassium sulfate, $FeSO_4$, $FeCl_3$ and PAC. The effect of process parameters, such as, initial pH (pH), coagulant dose and reaction time were observed.

2. Materials and methods

2.1. Effluents

The desizing wastewater samples were obtained from a textile mill located at Ghaziabad, U.P., India. The original effluent had a COD of 2884 mg/l and color 520 Pt-Co. In the present studies, the coagulation studies of the fresh (Effluent 1) and the thermally treated

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Nomenclature

A	filtration area (m^2)
BOD	biochemical oxygen demand (kg/m^3)
C	concentration of slurry (kg/m^3)
C_u	concentration of solids required in the underflow
C_0	initial solids concentration (kg/m^3)
COD	chemical oxygen demand (kg/m^3)
COD_0	initial conc. of organic matter in the effluent expressed as COD (kg/m^3)
PCU	platinum cobalt unit (Pt-Co)
pH	initial pH
pH_f	final pH
R_m	filter medium resistance (m^{-1})
RM	rapid mixing
SM	slow mixing
t	time (s)
t_R	treatment time (h)
T	absolute temperature (K)
v_f	volumetric flow rate (m^3/s)

Greek symbols

α	average cake resistance (m/kg)
μ	viscosity of the filtrate (Pa s)

desizing wastewater (Effluent 2) were done. The characteristics of the wastewaters are given in Table 1.

2.2. Chemicals

The chemicals used as coagulants were of analytical reagent grade. $FeCl_3$ and $FeSO_4{\cdot}7H_2O$ were obtained from Qualigens Fine Chemicals, Mumbai, India and PAC was obtained from Vam Organics Ltd., Gajraula, U.P., India.

2.3. Analytical methods

The COD of the effluent was estimated by the Standard Dichromator Closed Reflux Method (APHA-1989) using a COD analyzer (Aqualytic, Germany). The color in Pt-Co unit was estimated using a color meter (Hanna HI93727, Hanna Instruments, Singapore) at 470 nm and the pH was measured using a Thermo Orion, USA make pH meter. The treated wastewater samples were centrifuged (Model R24, Remi Instruments Pvt. Ltd., Mumbai, India) to obtain the supernatant and the precipitate.

Table 1
Characteristics of effluents

S. no.	Parameters	Effluent 1[a] Value (mg/l)	Effluent 2[b] Value (mg/l)
1.	Total suspended solids	56.7	–
2.	Total iron (as Fe)	2.37	–
3.	Chlorides (as Cl)	950	–
4.	BOD, 3 days at 20 °C	3275	72
5.	Oil and grease	145.9	
6.	Sulfate (as SO_4)	520.94	–
7.	Copper (as Cu)	0.038	–
8.	Manganese (as Mn)	0.64	–
9.	Zinc (as Zn)	0.364	–
10.	COD	2884	819
11.	Color	520 (PCU)	66.6 (PCU)

[a] Effluent 1: Fresh desizing wastewater.
[b] Effluent 2: Thermally treated desizing wastewater.

The FTIR spectra of the dried desizing wastewaters before and after treatment were recorded on a FTIR Spectrometer (Thermo Nicolet, USA, Software used: NEXUS) in the 4000–500 cm^{-1} wave number range using KBr pellets.

2.4. Jar test

A series of six graduated glass cylinders were used for the experiments. A 5 min rapid mixing (RM) at 80 rpm was followed by 30 min slow mixing (SM) at 40 rpm and 120 min settling. The objective of this experiment was to study the settling characteristics as well as estimation of COD and color in the final clear liquid [10]. The procedure comprised of the following steps. The runs at ambient temperature were taken in a 0.5 l beaker. The waste water was put into the beaker after adjusting the desired pH and at the same time the coagulant/chemical was added to the beaker. The reaction mixture was agitated for 30 min slower and 30 min rigorously using a paddle agitator and then the waste water was allowed to settle for 2 h. Thereafter a small amount of sample was taken out to determine the pH and COD of the treated sample. The solution left was again mixed rapidly for 5 min to make it a homogeneous solution and then, it was examined for the settling and filterability characteristics. The sludge was obtained after settling, decantation and drying of the treated effluent by coagulation/thermolysis.

3. Results and discussion

3.1. Effect of pH

The results of the variation of %COD reduction and %color reduction as a function of pH are presented in Figs. 1 and 2. The pH of the initial feed effluent were varied from 2 to 12, keeping the coagulant dose fixed at 3 kg/m^3 in each run. It has been generally observed that the percent reductions reach two maximums, one in acidic region and the other in alkaline region. The maximum COD reduction of 52.46% was observed using commercial alum at pH 4. This was followed by aluminum potassium sulfate (pH 4), $FeCl_3$ (pH 6), PAC (pH 6), $FeSO_4$ (pH 4) and without coagulant (pH 4). The reduc-

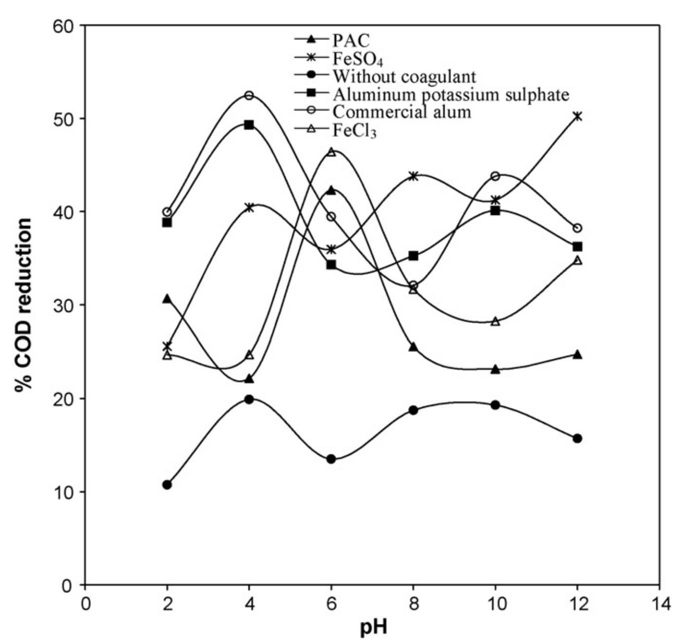

Fig. 1. Effect of pH on COD reduction of the desizing wastewater by using different coagulants. P = atmospheric pressure, COD_0 = 2884 mg/l, C_w = 3 kg/m^3.

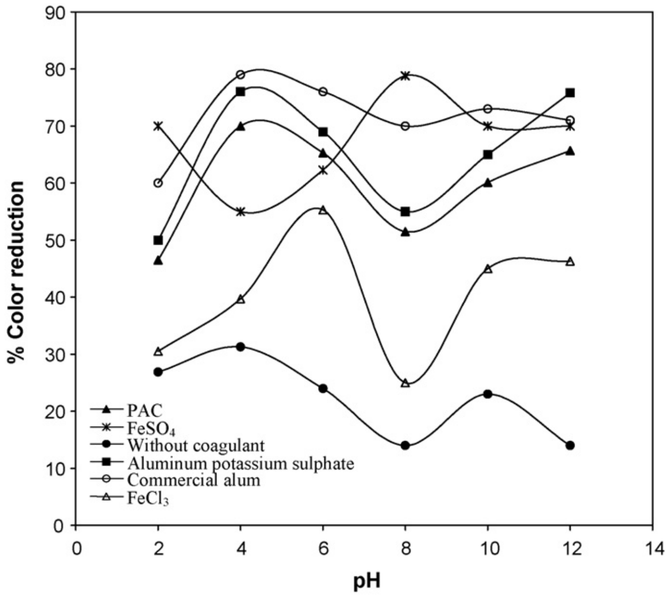

Fig. 2. Effect of pH on color reduction of the desizing wastewater by using different coagulants. P = atmospheric pressure, COD_0 = 2884 mg/l, C_w = 3 kg/m^3, initial color concentration = 520 PCU.

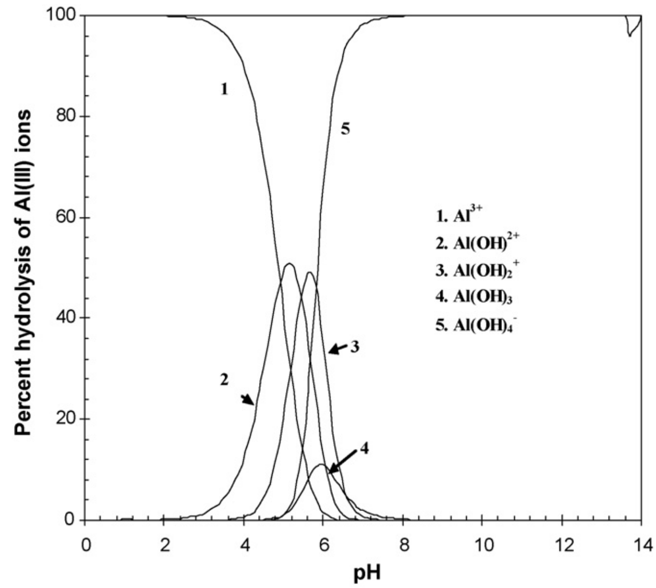

Fig. 3. Speciation diagram of Al(III) ion.

tion in COD obtained without the addition of a coagulant was 19.9% at pH 4. This was the maximum COD reduction observed due to variation in pH.

Similar trend was observed during variation in %color reduction as a function of initial pH. Commercial alum gave maximum color reduction of 79% at pH 4. This was followed by aluminum potassium sulfate (pH 4), PAC (pH 4), $FeSO_4$ (pH 2), $FeCl_3$ (pH 6) and no coagulant (pH 4).

The effect of pH on the COD and color reduction could be explained by the combined effect of (i) the ionization of amino, hydroxy and sulfo groups in the dye molecules present in the desizing wastewater which increases with pH in acidic range, (ii) the decrease in the concentration of dissolved hydrolysis products and (iii) Al-based coagulants show better results than that of Fe-based coagulants. This may be attributed to dominance of compounds having positive charge. The cationic compounds of the hydrolyzed dyes act as good reagents and electron acceptors from Al^{3+}. Thus, the complexation followed by precipitation and the capture of organic in the gel are responsible for both COD and color reduction by commercial alum as compared to those obtained with $FeSO_4$ and $FeCl_3$.

The reduction of dissolved organics during coagulation with metal salts at different pH values follow two different mechanisms. At low pH, the effluent containing anionic organic molecules coordinate with metal cation and form insoluble metal complexes at higher pH (alkaline range). The organics are adsorbed on to preform flocs of metal hydroxides and get precipitated. The combined effect of two mechanisms is that the reduction of dissolved organics with different functional groups can occur at different pH. The maximum COD and color removal may thus occur at a pH where the combined effect of both the mechanisms is high.

Aluminum species are found to be present in deionized water in the form of Al^{3+}, $Al(OH)^{2+}$, $Al(OH)_2{}^+$, $Al(OH)_{3(S)}$, $Al(OH)_4{}^-$, etc. In tap water, the species may be carbonate, bicarbonate, sulfate, chloride, etc. depending upon the presence of these anions in the water. The concentration of the hydrolyzed aluminum species depends on the aluminum concentration, and the solution pH. The speciation of Al(III) ions in deionized water is presented in Fig. 3. The percentage of Al^{3+} hydrolytic products was calculated from the following stability constants [11]:

$$Al^{3+} + H_2O \rightleftarrows Al(OH)^{2+} + H^+ \quad pK_1 = 4.95$$

$$Al(OH)^{2+} + H_2O \rightleftarrows Al(OH)_2{}^+ + H^+ \quad pK_2 = 5.6$$

$$Al(OH)_2{}^+ + H_2O \rightleftarrows Al(OH)_3 + H^+ \quad pK_3 = 6.7$$

$$Al(OH)_3 + H_2O \rightleftarrows Al(OH)_4{}^- + H^+ \quad pK_4 = 5.6$$

The natural pH of the desizing wastewater is 4.0 (Table 1). The adsorption of natural organic matter present in the wastewater on the colloids will give a negative surface charge [11]. It is evident from Fig. 3 that at pH ~ 4.0, Al^{3+} is the main component of aluminum present in the water. It is predicted that this metallic anion gets adsorbed onto the negatively charged cations causing charge neutralization. This may allow different colloids to come together and enhance the COD and color reduction at pH ~ 4.0. Heterocoagulation may also explain this behaviour. Heterocoagulation occurs between two different types of particles. When the two particles have different charges, both the van der Walls and the electrostatic forces act and this provide the conditions for an effective coagulation [12].

3.2. Effect of coagulant dose (C_w)

During coagulation, the effluent is gently mixed which initiates floc formation, complexation and adsorption of organics resulting in precipitate formation and settling down of insoluble solids. Commercial alum was found to be the best coagulant amongst the various coagulants used. In order to determine the optimum commercial alum dose, the effect of coagulant dose was studied in the range of 1–7 kg/m^3 at optimum pH 4. Fig. 4 shows the results for COD as well as color reduction. It is observed that the COD and color reduction increase linearly with increase in coagulant dose from 1 to 5 kg/m^3. The rising trend, however, do not continue beyond 5 kg/m^3, getting almost no increase in the reduction of both the parameters. The maximum COD reduction observed at a coagulant dose of 5 kg/m^3 was 58.34% whereas the color reduction at these conditions was 85%.

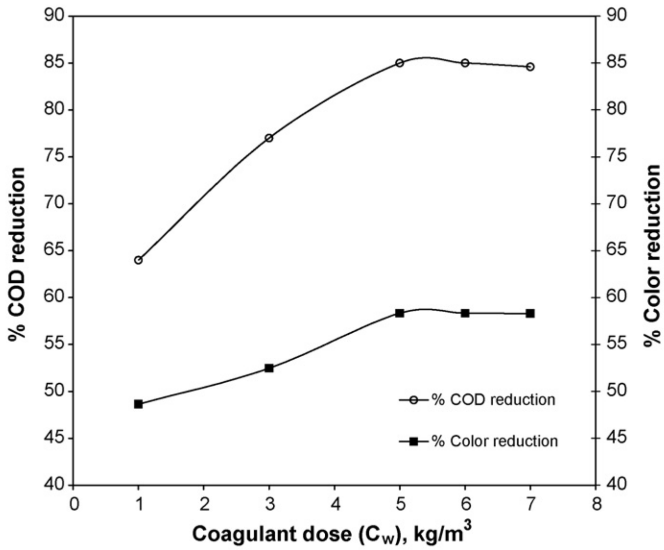

Fig. 4. Effect of coagulant (commercial alum) dose on COD and color reduction of the desizing wastewater. P = atmospheric pressure, COD_0 = 2884 mg/l, pH 4, initial color concentration = 520 PCU.

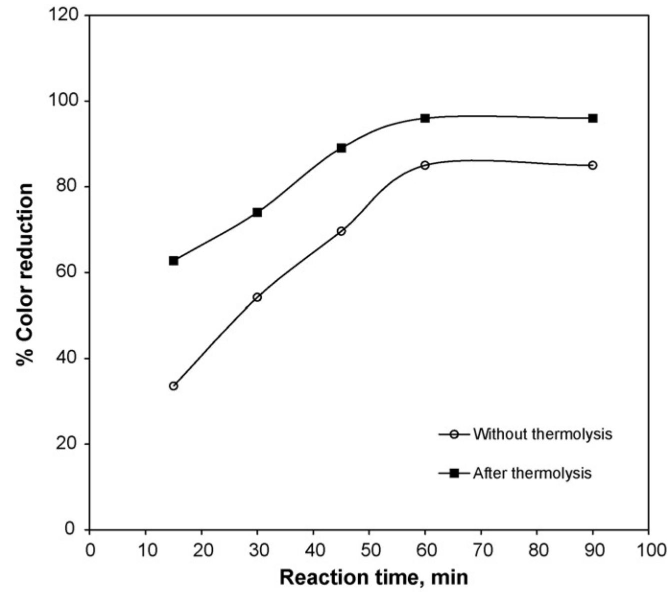

Fig. 6. Reduction of color with time by coagulation using commercial alum as coagulant. P = atmospheric pressure, COD_0 = 2884 mg/l, pH 4, T = 18 °C, initial color concentration = 520 PCU.

3.3. Catalytic thermolysis followed by coagulation

Figs. 5 and 6 show the effect of time for the COD and color reduction of desizing wastewater (COD_0 = 2884 mg/l) and thermally treated desizing wastewater (COD_0 = 819 mg/l), respectively. It is observed that coagulation of the supernatant obtained after catalytic thermolysis is more effective in treating the effluent than the individual processes. The figures demonstrate that a reaction time of 60 min is enough to achieve the steady state operation.

The application of coagulation to the supernatant obtained after thermolysis show a removal of 87.96% COD and 96.0% color at above mentioned conditions except at a coagulant dose of 1 kg/m^3. The amount of inorganic sludge generated gets drastically reduced (almost 25%) due to the reduced amount of coagulant. The COD and color of the final effluent were found to be 98.6 mg/l and 2.67 PCU, respectively, and the COD/BOD_3 ratio was 1.36.

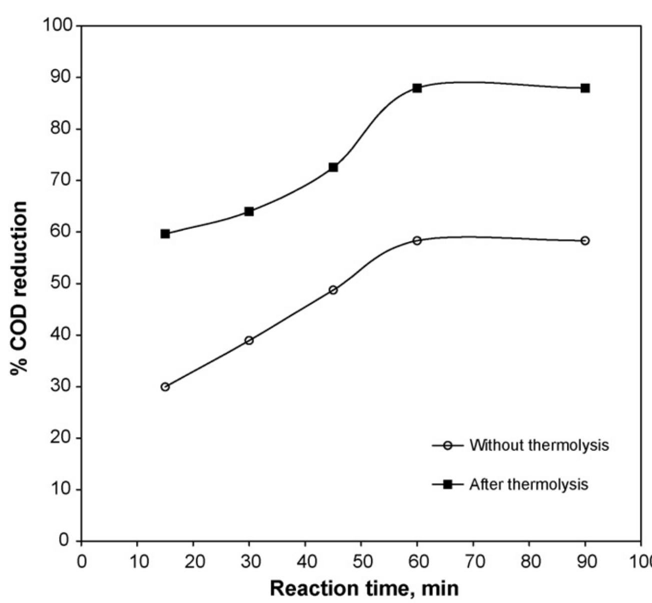

Fig. 5. Reduction of COD with time by coagulation using commercial alum as coagulant. P = atmospheric pressure, COD_0 = 2884 mg/l, pH 4, T = 18 °C.

IR spectral studies of waste samples (before and after treatment) provide useful information on the presence of different functional groups. For example, desizing wastewater sample exhibits a broad band covering the region 3300–2800 cm^{-1} suggesting the presence of hydrogen bonded ν(OH) group. A broad band at ~1600 cm^{-1} due to δ(OH) further suggest the presence of hydroxyl group (Fig. 7a). A strong band at 1124 cm^{-1} confirms the presence of sulfate group possibly attached with metal ions. In addition, the presence of C=C (conjugated carbon-carbon bond) is indicated by the presence of medium intensity band at 1582 cm^{-1}.

Dried sludge obtained after treatment with $CuSO_4$ gave relative sharper but still broader band at ~3400 cm^{-1}. This is possibly due to breaking of hydrogen bond and presence of either free hydroxyl or coordinated hydroxyl group. This also suggests the precipitation of component bearing hydroxyl group present in either complexed or free form. Absence of band due to sulfate group in this component indicates the removal of sulfate group as water soluble component. The band at 1086 cm^{-1} is possibly due to the presence of metal oxide, which is further supported by the appearance of two bands at 619 and 461 cm^{-1} due to ν(M–O) (Fig. 7b).

A broad band appearing in the 3400 cm^{-1} region in dried desizing wastewater (Fig. 7c) suggests the presence of ν(OH) due to the presence of hydroxyl groups from different components. Treatment with commercial alum and copper sulfate showed a decrease in the ν(OH) peak in the spectra. This decrease in broadness indicates the precipitation of a particular type of component. The band covering the region 2850–2924 cm^{-1} in dried desizing waste as well as solid residue after the treatment indicates the presence of $-CH_2$ group.

A medium intensity band at 1552 cm^{-1} is assigned to C=C conjugate bond. This bond is slightly shifted to higher wave number due to adjustment of current after coordination of part of the –OH group with metal. The presence of two bands at 1680 and 1450 cm^{-1} in the dried sample are due to antisymmetric and symmetric modes of carboxylic acid. It is expected that glucoisosacchararinic acid having carboxylic group may be present in the cellulose derivatives. The Δ(antisym − sym) value of 230 cm^{-1} suggests the monodentate coordination of carboxylate group with metal salt on treatment.

Presence of sulfate group as a sharp band still appears at 1109 cm^{-1} due to ν(S=O). Interestingly, the bands appearing at 2341 and 2360 cm^{-1} in both of the above wastes/sludges are absent suggesting either the conversion of this group to other components or their removal during treatment.

3.4. *Settling characteristics of the precipitate in the treated effluent*

The settling characteristics of the desizing wastewater after coagulation with commercial alum at pH 4 and 12 at ambient tem-

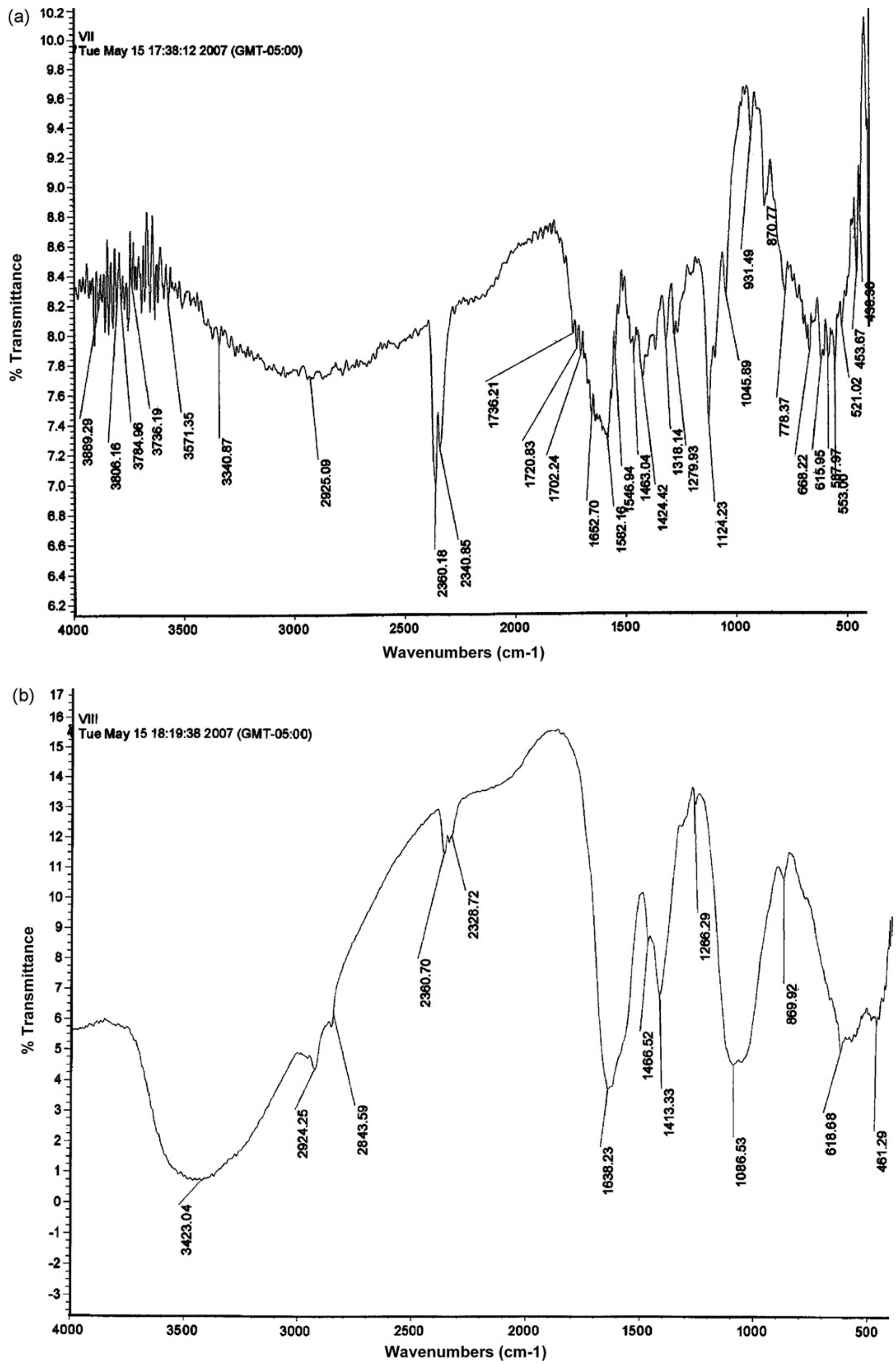

Fig. 7. (a) FTIR spectra of desizing waste water (dried). (b) FTIR spectra of desizing waste water after catalytic thermolysis (dried). (c) FTIR spectra of desizing waste water after coagulation (dried).

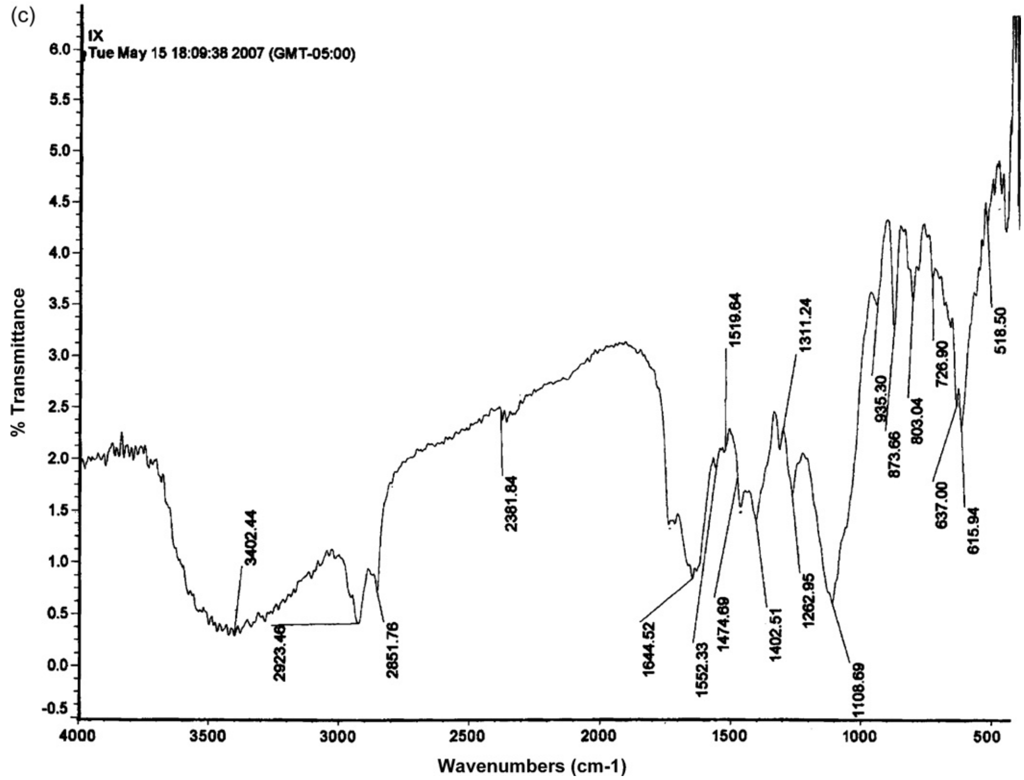

Fig. 7. (*Continued*).

perature 20 °C was observed in a 100 ml measuring cylinder. This was done to see the effects of the pH on the settling characteristic of the precipitate. The settling rate was observed to be higher for pH 12 than that of pH 4, probably due to the bigger size and more compact aggregated flocs. Fig. 8 shows the behaviour of treated effluent during sedimentation.

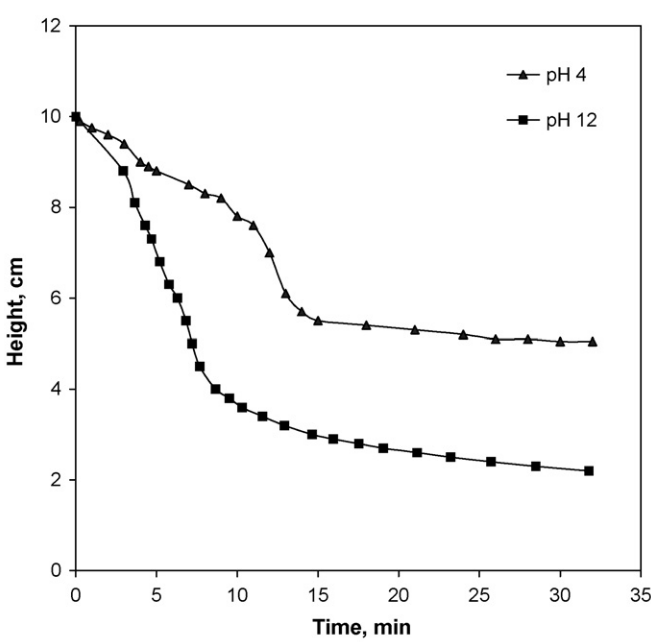

Fig. 8. Settling characteristics of desizing wastewater after coagulation at different pH using commercial alum as coagulant. $COD_0 = 2884$ mg/l, P = atmospheric, $C_w = 5\ kg/m^3$.

Although the average driving forces in batch and continuous sedimentation operations are different and the batch driving force varies with time, approximate methods are available for the calculations of the compression zone depth in continuous thickeners [13–16]. Although a number of papers have appeared in the literature on the subject of sedimentation [17–19], it is still preferable to use the method proposed by Richardson et al. [14] to design a continuous thickener based on single batch sedimentation test was used for calculating compression zone height in continuous thickeners from the batch sedimentation data. The method of Richardson et al. [13] gives a conservative estimate with an inherently high safety limit due to the changing nature of the flocs and their settling and compression characteristics.

The parameters such as sedimentation velocity (u_c), concentration $C(t)$, and the sedimentation flux were calculated. The sedimentation velocity was found as the slope of the tangent at a given solid concentration C. The concentration of sludge at a time t was determined by using the following formula $C = C_0$ (total height)/(height of suspension after time t). The area of the sedimentation tank for any effluent flow rate can thus be calculated as

$$A = v_f \frac{C_0[(1/C) - (1/C_u)]}{u_c}$$

where v_f is the volumetric flow rate of the effluent (m^3/s), C_0 the initial solid concentration (kg/m^3) and C_u is the concentration of solids required in the underflow.

The settling of the effluent treated at a higher pH is faster than that treated at a lower pH. Thus, an increase in the treatment temperature will bring down substantially the area of the sedimentation tank. From Fig. 8, it can also be seen that the settling rate is much faster during the zone settling region at pH 12. The settling rate becomes very slow, as the solids settling enter compression region. It is also seen that the compression region for the pH 12

Table 2
Filterability of the slurry of desizing wastewater after treatment: effect of the initial pH

pH	$k_p \times 10^{-10}$ (s/m^6)	$\beta \times 10^{-6}$ (s/m^3)	C (kg/m^3)	$\mu \times 10^3$ (Pa S)	$\alpha \times 10^{-10}$ (m/kg)	$R_m \times 10^{-8}$ (m^{-1})
4	0.833	15	4.5	1.96	55.35	70.55
12	0.348	2	4.7	1.98	21.91	93.12

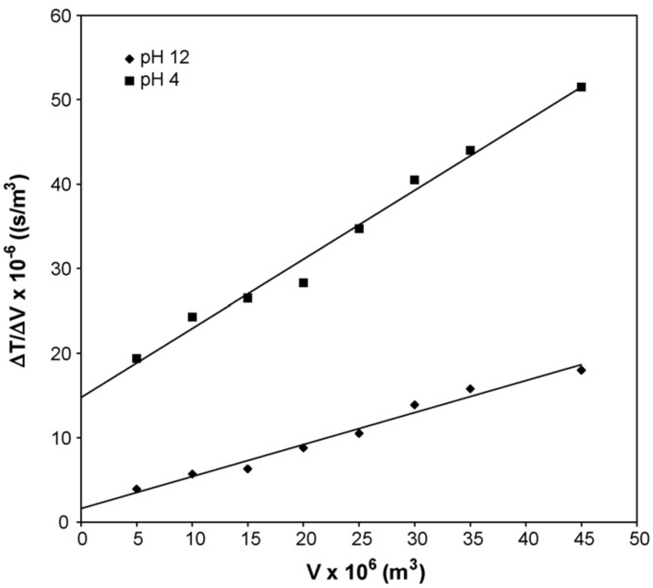

Fig. 9. Effect of initial pH on the filterability of the desizing wastewater after coagulation. COD_0 = 2884 mg/l, P = atmospheric pressure, C_w = 5 kg/m^3.

settled sludge is much denser (more than twice) than that for the pH 4 settled sludge.

3.5. Filterability study

Because of the treated effluent is to be separated from its residue and the liquid effluent is to be treated further, it was necessary to test the gravity filtration characteristics of the slurry at ambient temperature on an ordinary filter paper supported on a Büchner funnel. The ceramic Büchner funnel diameter was 0.15 m. The change in the hydrostatic head was assumed negligible, and the gravity filtration was considered as constant pressure filtration. The filtrate volume with time were observed, and a plot between $\Delta t/\Delta V$ and V was drawn for the effluents treated at different pH. The filtration resistances for the filter media as well as the filter cake were obtained using the filtration equation [20]:

$$\frac{dt}{dV} = k_p V + \beta \quad (1)$$

$$\text{where } k_p = \frac{C\alpha\mu}{A^2(-\Delta p)} \quad (2)$$

$$\text{and } \beta = \frac{\mu R_m}{A(-\Delta p)} \quad (3)$$

A plot of Eq. (1) for the experimental data is presented in Fig. 9 from which the values of k_p (slope) and β (intercept) were determined. The value of α and R_m were calculated from k_p and β and are presented in Table 2.

The viscosity of the filtrate was determined at room temperature (18 °C) using an Ostwald capillary viscometer. It is observed that pH has a pronounced impact on the filterability of the desizing wastewater after coagulation.

Typical values of specific cake resistance for different sludge's are given by Barnes et al. [21]. Pulp and paper mill effluent characteristics are given by Garg et al. [22], alcohol distillery waste characteristics are presented by Lele et al. [23] whereas composite and dyeing wastewaters are presented by Kumar et al. [7].

The reported values for other effluents show variation than those shown in Table 2 for desizing wastewater. The difference can be ascribed to several factors like treatment conditions, morphological and floc characteristics of the sludge, which may be different for the textile mill effluent.

4. Conclusion

The present study deals with treatment of fresh desizing wastewater and thermally treated desizing wastewater using different coagulants, such as, commercial alum, aluminum potassium sulfate, $FeSO_4$, $FeCl_3$ and PAC. The maximum COD reduction observed at a coagulant (commercial alum) dose of 5 kg/m^3 and pH 4 was 58.34%, whereas, the color reduction at these conditions was 85%. The application of coagulation on clear liquid obtained after catalytic thermal treatment is most effective in removing nearly 88% of COD and 96% of color at above mentioned conditions except at a lower coagulant dose of 1 kg/m^3 The amount of inorganic sludge generated, thus, gets drastically reduced due to the reduced amount of coagulant. The COD and color of the final effluent were found to be 98.6 mg/l and 2.67 PCU, respectively, and the COD/BOD_3 ratio was 1.36. The settling rate of the slurry was found to be strongly influenced by treatment pH. The slurry obtained after treatment at pH 12 settled much faster in comparison to slurry obtained at pH 4. The filterability of the treated effluent is also strongly dependent on pH. pH 12 was adjudged to be the best in giving highest filtration rate.

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