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Removal of color and chemical oxygen demand from sugar industry wastewater using thermolysis processes

O.P. Sahu, P.K. Chaudhari*

Department of Chemical Engineering, National Institute of Technology, Raipur, India, Tel. +91 097526 10957; email: ops0121@gmail.com (O.P. Sahu), Tel. +91 771 2254200, +91 094061 45088; email: pkchaudhari.che@nitrr.ac.in (P.K. Chaudhari)

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ABSTRACT

In the present study, treatment of sugar industry wastewater (SIWW) was investigated for the reduction of chemical oxygen demand (COD) and color using catalytic thermal treatment (thermolysis). Effect of various parameters such as pH 2–10, temperature 55–95°C, catalyst mass loading (C_w) 2–5 kg/m^3, and types of catalysts was studied on COD and color reductions of the effluent. Among various catalysts used, copper oxide was found to be the best catalyst giving 74% COD and 80% color reductions with a catalyst concentration of 4 g/dm^3 at pH 10 and 75°C temperature. Order of COD degradation rate was found to be 1.5 with respect to COD and 0.805 with respect to catalyst mass loading. Settling rate of the treated effluent was good and was found to be affected by the treatment temperature. The initial pH (pH$_i$) of the effluent played an important role in the treatment process, and optimum value was found to be different for different catalysts. Results show that the process can be applied for the treatment of SIWW.

Keywords: Catalyst; COD reduction; Filtration; Sugar industry wastewater; Settling; Thermolysis

1. Introduction

Wastewater from sugar industries has complex characteristics, and its treatment is crucially important not only for the environment point of view but also for the purpose of recycling of water for further use in industrial processes. A sugarcane-based industry generated effluent contains chemical oxygen demand (COD) 2,300–8,000 mg/dm^3, BOD 1,700–2,300 mg/dm^3, and suspended solids up to 5,000 mg/dm^3 [1]. Apart from these, it contains oil, greases, and pathogens. These pollutants have to be removed up to the accepted norms as set by pollution control agencies. The norms usually depend upon whether the water will be reused or discharged into a receiving stream. Available wastewater treatment processes can be broadly classified as physicochemical, biological, and advance treatment. These processes consist of a series of unit operations, applied in different combination and sequence depending upon the prevailing situations of effluent concentration, compositions, condition and specification. In sugar industries, the wastewater stream generated from different sections of plant is taken in equalization tank which is further treated using options such as: (i) anaerobic digestion to produce methane gas followed by bio-aerobic treatment and (ii) coagulation using lime and alum followed by bio-aerobic treatment. Both the processes are not able to bring the effluent to the

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*Corresponding author.

wastewater discharge standard for release into surface water (COD < 100 mg/dm^3) and sewers (COD < 300 mg/dm^3) prescribed by the World Health Organization.

Some processes have been reported for the treatment of sugar industry wastewater (SIWW). These include electrochemical oxidation [2,3], membrane separation [4], and biochemical oxidation [5]. No work has been reported for the thermal treatment of SIWW. In the recent years, thermolysis process has been investigated so as to treat moderate- to high-strength organic wastewaters emanating from pulp and paper mills [6], alcohol distilleries [7], textile industry [8], and petrochemical industries [9]. The temperature, pH, autogenous pressure, and catalysts have been found to be the key parameters affecting the thermolysis process. Thermolysis is the process of chemical transformation of the dissolved organics and inorganics into suspended/settleable forms with or without metal compounds (salts) at moderate temperature and self (autogenous)-pressure. At low temperature, thermo-chemical precipitation of dissolved organic and inorganic salts takes place, which results in the removal of COD, BOD, and color of wastewater. Inorganic salts hasten the process of metal complexation and precipitation, just as coagulants/flocculants work at room temperature. The organics present in the wastewater either get polymerized or decomposed into smaller molecules or get complexed with metal salts at 60–300°C.

Sugar industry generates 1 m^3 of wastewater per ton of cane processing which contains a high amount of pollution load, particularly in terms of organic matter. Therefore, an attempt has been made to treat the effluent generated by sugar industry located in Kawardha, India. The aim of the present study is to find the effectiveness of catalytic thermolysis process to treat SIWW. Effects of various parameters such as pH, type of catalysts, catalyst mass loading, and reaction time on treatment of the effluent have been presented. To separate the solid and liquid of the slurry, the filtration and settling process were also performed.

2. Materials and methods

2.1. Materials

The wastewater was arranged from Bhoramdev Sugar Industry Ltd. Kawardha, Chhattisgarh, India. It was kept at 4°C in the freezer to prevent its quality from any change. The laboratory grade chemicals made by Merck India Limited, Mumbai, were used for thermolysis experiments; however, for analysis, only analytical grade chemicals were used.

2.2. Experimental setup and procedure

A Borosil glass reactor 0.5 dm^3 capacity was used for the thermolysis experiments at atmospheric pressure. The rector equipped with a proportional integral derivative controller (PID) temperature indicator cum controller, a long vertical condenser for condensing the vapor, and a magnetic stirrer with variable speed regulator was used in the experiments. The experimental setup is presented in Fig. 1.

For thermolysis experiment, 0.3 dm^3 SIWW was taken in glass reactor. The desired amount of catalyst was taken in it and was preheated from the ambient temperature (T_0) to the treatment temperature (T_R). The preheating period (θ) was varied with the T_R. Therefore, the time of the start of treatment was considered as the "zero time" when the T_R was attained after the preheating of the wastewater from T_0. Five milliliters of the sample were withdrawn at a definite interval of time and allowed for settling of sludge. After this, the supernatant was analyzed for its COD and color. The initial pH of the wastewater was varied between 2 and 10 using either 0.5 N HCl or 1 M NaOH.

2.3. Catalyst preparation

$CuSO_4$, $FeSO_4$, CuO, and MnO were used as catalysis for thermolysis process. $CuSO_4$ and $FeSO_4$ were used as obtained. The metal oxide catalysts, such as CuO and MnO, were prepared by alkali precipitation from an aqueous solution of a metal salt followed by drying and calcination. The weighed samples were dissolved in distilled water, and liquid ammonia was slowly added dropwise to the metal salt solution with stirring. The resulting mixture was filtered, and the retained precipitate on the filter paper was

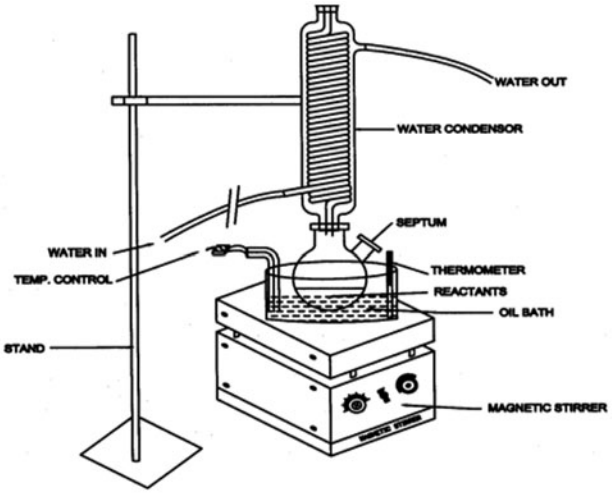

Fig. 1. Experimental diagram of equipment.

double-washed with distilled water to remove the traces of alkali present in the sample. The washed precipitate was removed from the filter and dried in an oven at 105℃ for 18 h, which was followed by calcination at 350–400℃ for 4 h. The calcined catalyst was grounded in a laboratory grinder and sieved. The solid particles having an average size of 220 μm were used in the experiments.

2.4. Analytical procedure

The COD of the samples was determined by the standard dichromate open reflux method [10]. The chloride concentration was determined by the standard titrimetric Volhard method [11]. Sulphate contents were determined using gravimetry. Phosphates were determined using standard methods [10]. The protein content was determined by the Lowery method [12]. The color of the sample was measured in terms of the absorbance at $\lambda = 420$ nm [13] using a UV–vis spectrophotometer.

3. Results and discussion

3.1. Effect of pH on COD and color reductions

Thermolysis process has been found to be highly dependent on pH of the solution for the treatment of distillery wastewater [7], pulp and paper mill effluent [6], and textile industry wastewater [8]. Therefore, the effect of pH_i on treatment of SIWW was studied. The process was performed in the AGR at atmospheric pressure and 75℃ for 4 h using a catalyst mass loading of 4 g/dm^3 effluent. Initial pH (pH_i) of wastewater was varied in the range of 2–10. After each experiment, a portion of the treated effluent was taken out and sludge was allowed to settle. The clear liquid was taken, and its COD as well as color was measured. The results of COD and color reductions are shown in Fig. 2(a) and (b). The highest COD reduction of 60.3% was obtained with CuO catalyst at pH 10. COD reduction with CuO catalysis was found to increase from pH_i 2–4, then decreased at pH 6, and further increased up to pH 10. With MnO catalysis, COD reduction continuously increased with the increase in pH up to 8 (56.5% COD reduction) and decreased slightly at pH 10. With $CuSO_4$ catalysis, the highest COD reduction was obtained at pH 4 (49%), and after this, it decreased marginally with the increase in pH. Similarly, decreasing trend of COD reduction, but slight increase in pH_i, was seen with $FeSO_4$ catalyst. In the absence of catalyst, COD reduction was least at pH 2 (23%), which increased marginally with increase in pH_i to 38% COD reduction at pH 10. Almost similar trend was observed for color reduction. The highest color reduction of 72% was observed for CuO catalyst at pH 10 which was followed by 64% at pH 8 with MnO, 60% at pH 4 with $CuSO_4$, 54% at pH 4 with $FeSO_4$, and 41% at pH 10 without catalyst. This type of random COD reduction values were also observed for treatment of various effluents by previous investigators [6–8]. SIWW contains carbohydrate, protein and melanoidin which have different functional groups. The COD reductions of effluent were due to the combined effect of an active functional group of effluent which reacts at certain pH with the catalysts. Formations of solid residues by thermo-chemical precipitation as well as by hydrolysis of organics to carboxylic acids are the mechanisms responsible for COD and color reduction of the effluent.

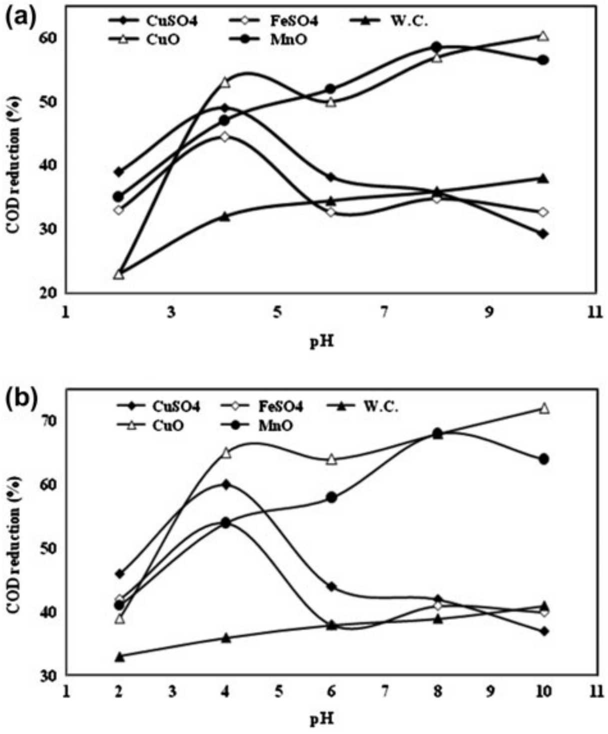

Fig. 2. Effect of pH on COD and color reduction using various catalyst. C_w = 4 kg/m^3 temperature 75℃.

3.2. Effect of reaction time on COD and color reductions

Reaction time is another significant parameter which influences the thermolysis process. Formation of solid complex as well as breaking of larger molecules to smaller molecules, thus reduction of pollutants, depends on reaction time. The effect of reaction time on treatment efficiency was analyzed at optimum pH_i, 75℃, and 4 kg/m^3 catalyst loading. COD and color reductions are shown in Fig. 3. It was observed that the

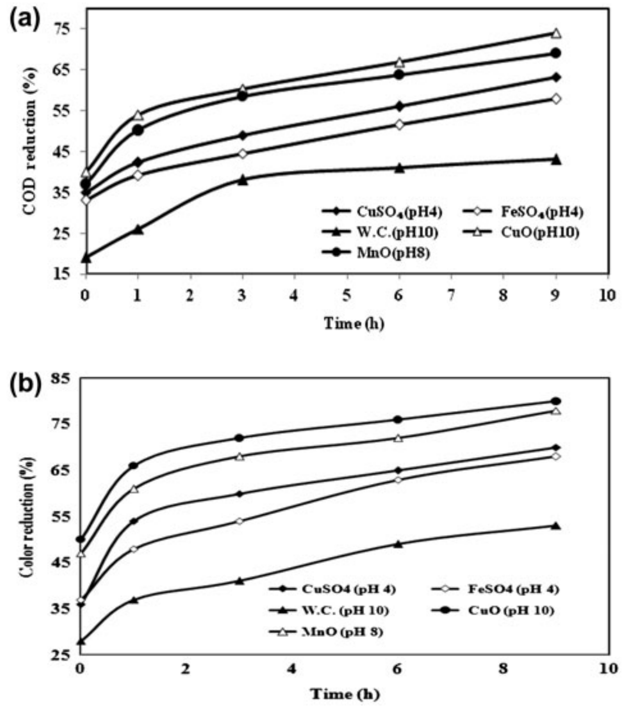

Fig. 3. (a) COD and (b) color reduction with time at catalyst optimum pH, temperature, 75˚C, and $C_\mathrm{w} = 4\ \mathrm{kg/m^3}$.

efficiency increased with the increase in reaction time. From Fig. 3, it may be seen that CuO gave the maximum 74% COD and 80% color reductions in 9-h treatment time. At the beginning of the reaction when the reaction time is zero, that is, time when the temperature reached from room temperature to reaction temperature, the COD and color reductions were 40 and 50%, respectively. MnO was found to work better in terms of COD (69%) and color (78%) reductions as compared to $FeSO_4$, for which removal of 58% COD and 68% color was obtained in 9 h. Lowest COD reduction (43%) and color reductions (53%) were seen when SIWW was treated without catalyst. The phenomenon of reduction in COD and color during the heating period in the absence of oxygen was because of the breaking of large molecules into small molecules by thermal decomposition as well as because of the formation of solid mass which settled down. At preheating time and initial periods of thermolysis (up to 4 h), high COD and color reductions are due to the availability of fresh catalyst and relatively high reactants. Reactant amount decreased with time, and catalyst fouled with time.

3.3. *Effect of catalyst mass loading*

The effect of copper oxide mass loading on the COD and color reductions of the SIWW was observed at a temperature 75˚C. The copper oxide mass loading was varied from 2 to 6 g/dm^3, and pH was kept 10 for all the experimental runs. Only 62% COD and 64% color reductions were obtained with 2 kg/m^3 of catalyst mass in 9 h of experiments. With further increase in mass loading to 4 g/dm^3, a maximum of 74% COD and 80% color reductions were obtained, as shown in Fig. 4. Beyond 4 g/dm^3 catalyst mass loading, almost no increase in the COD and color reductions was observed. For 5 g/dm^3 catalyst, 73% COD reduction and 77% color reduction were noted. Thus, 4 g/dm^3 catalyst mass loading is the optimum concentration of catalyst. Similarly, almost no increase in COD reduction was observed in previous studies when catalyst mass loading was increased from an optimum dose for thermal treatment of organic effluents [6–8]. The final pH of the treated wastewater decreased from its initial value attributed to formation of carboxylic acids (Table 1).

3.4. *Effect of temperature*

To find the effect of temperature in the process, experiments were conducted in the temperature range of 55–95˚C, pH_0 10 and CuO catalytic loading (C_w)

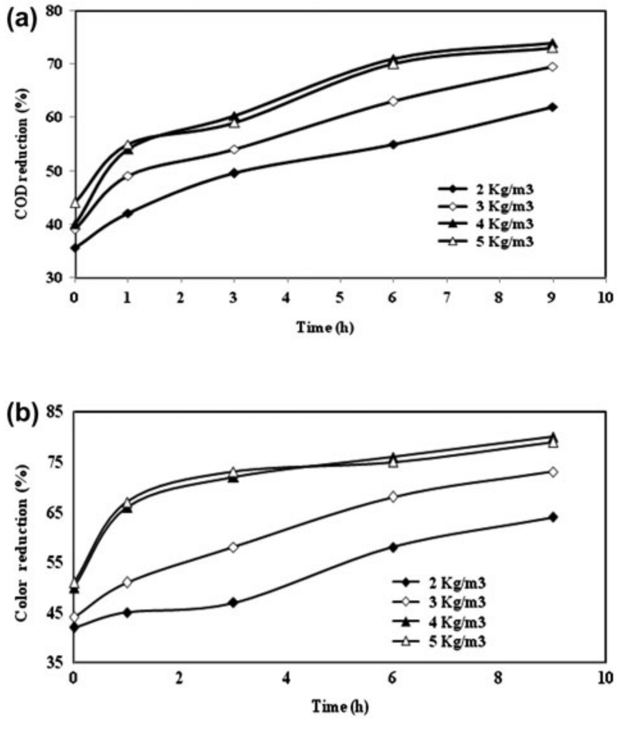

Fig. 4. Effect of catalyst mass loading on (a) COD and (b) Color reduction using CuO catalyst. pH 10 and temperature 75˚C.

Table 1
Characteristic of SIWW and thermally treated SIWW. CuO 4 kg/m^3 and pH 10

S. No.	Characteristics	SIWW	95°C	85°C	75°C	65°C	55°C
1	Color	Dark yellow	Light green	Light green	Light green	Light green	Light green
2	pH change	5.5	5.8	5.6	5.4	5.4	5.1
3	COD (mg/dm^3)	3,682	1,112	1,013	957	1,207	1,473
5	Phosphate (mg/dm^3)	5.9	4.8	4.3	3.5	4.6	5.2
6	Protein (mg/dm^3)	43	35	33	22	31	38
7	Total solid	1,940	645	657	620	668	715
8	Suspended solid	540	225	320	280	258	325
9	Dissolved solid	1,447	420	337	340	410	390
10	Chloride (mg/dm^3)	50	32	29	24	34	39
11	Hardness (mg/dm^3)	902	232	176	152	172	212

4 g/dm^3 for reaction time 9 h. As shown in Fig. 5, the percent reductions of COD and color increased as the temperature increased from 55 to 75°C. The maximum COD reduction of 74% and color reduction of 80% were obtained at 75°C with catalyst mass loading of 4 kg/m^3. After that, with an increase in temperature to 85 and 95°C, COD reduction decreased to 72.5 and 70% and color reduction to 77 and 74%, respectively. Since the experiments were conducted at atmospheric pressure (in a glass reactor under reflux), the reaction temperature was not exceeded beyond 95°C. The reason for the decrease in COD reduction with an increase in temperature may be due to the decomposition of solid residues at high temperature. Garg et al. [6] observed no increase in COD reduction with an increase in temperature in the range of 20–95°C when pulp and paper mill effluent was treated using CuSO$_4$ catalyst. During thermal treatment of desizing wastewater, Kumar et al. [8] found increase in COD reduction with temperature had 28 and 72% COD reductions at 60 and 95°C, respectively, with 4 kg/m^3 CuSO$_4$ catalyst. In our case, COD and color reductions are more. However, no work has been reported to treat SIWW by thermolysis process previously.

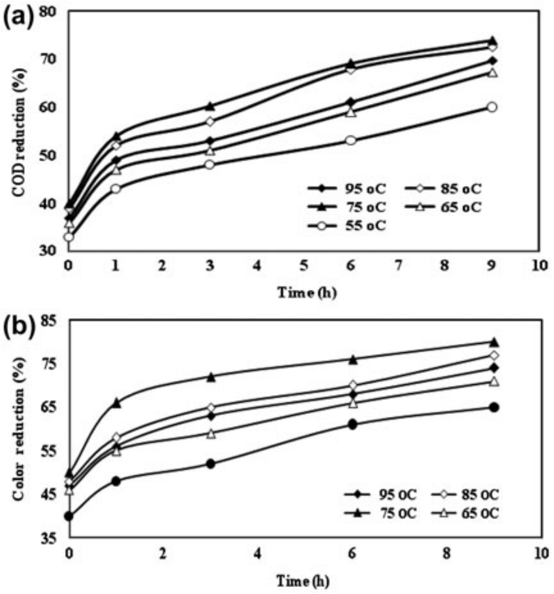

Fig. 5. Effect of reaction temperature on (a) COD and (b) color reduction. pH 10 and CuO catalyst C_w = 4 kg/m^3.

3.5. Thermolysis process

Coagulation process works best at low temperature, while thermolysis at high temperature. The organic wastewater contains various substances such as carbohydrates, proteins, lignin, melonidin, and other color-producing materials that depend on the raw material and process used. These components present various functional groups such as hydroxyl, carbonyl, and methoxyl groups, which take part in reaction, and their activity varies with pH. Catalysts' performance also depends on pH [7]. In thermolysis process, two mechanisms, both in parallel, but complementary to each other, take place simultaneously. Organic molecules present in the effluent undergo chemical and thermal breakdown and complexation forming insoluble particles which settle down in the reactor. Further, in the process, larger molecules also undergo breakdown into smaller molecules, which are soluble. Catalyst accelerates these actions. Metal cations such as Cu^{++} and Fe^{++} make complexation with organic molecules as well as they promote complexation between organics. In the present study, when the process was stopped, solid residues were obtained at the bottom of the reactor. Some soluble organics also get formed which are responsible for residual COD in treated liquor. Good heating values of residues were obtained from the treatment of pulp and paper mill effluent, distillery effluent, and textile mill effluent as reported by investigators [6–8]. The solid residue was found to have good settling characteristics.

3.6. Kinetics studies

The analysis of SIWW and treated effluent (Table 1) shows the presence of protein, dissolve solids, suspended solids, etc. These contribute to COD of the effluent. After treatment, their amount decreased. During the treatment under anoxic (absence of oxygen) conditions, the components of SIWW go through various reactions and produce highly reducing soluble and insoluble materials. It has been reported that soluble lignin, carbohydrates, melonidin, and proteins undergo various reactions forming soluble and insoluble residues [14–16].

In the presence of catalyst, the process of reduction of these compounds is accelerated. Since various organic components are present in SIWW, the rate equation may be based on the lumped reactions and the lumped concentration of all organic pollutants in industrial wastewater streams, that is, COD or total organic carbon (TOC) in wastewater. The COD reduction in the SIWW by the process can be represented as

$$\text{Sugar industry wastewater (organics)} \xrightarrow{\text{heating}} \text{Liquid organics} + \text{Solid residues} \tag{1}$$

It is further assumed that the inorganics do not contribute to the COD load of the effluent. COD (organic) reduction increases due to the increase in solid residue formation. The reduction equation can be written as

$$\text{COD} \xrightarrow{\text{heating}} \text{Liquid organics} + \text{Solid residues} \tag{2}$$

where the organic compounds in the wastewater are lumped as a single compound COD. The global rate expression for COD reduction due to the catalytic thermolysis taking into consideration the variation in catalyst mass loading can be expressed as homogenous-phase power-law equation:

$$-\frac{\mathrm{d}}{\mathrm{d}t}\text{COD} = k_n \text{COD}^n C_{\mathrm{w}}^m \tag{3}$$

For constant mass loading of catalyst, Eq. (3) can be further reduced to:

$$-\frac{\mathrm{d}}{\mathrm{d}t}\text{COD} = k\text{COD}^n \tag{4}$$

where

$$k = k_n {C_{\mathrm{w}}}^m \tag{5}$$

At the constant catalyst mass loading of 4 kg/m^3 and temperature 55–95˚C, Eq. (4) was solved numerically using Euler's integration method and fitted well for 1.5 order with respect to COD. The experimental values are shown by point and calculated values by line in Fig. 6. The values of k were evaluated to be 0.001115, 0.00155, 0.002, 0.0019, and 0.0016 dm$^{1.5}$/(mg$^{0.5}$ h) at the temperature 55, 65, 75, 85, and 95˚C, respectively. The values of rate constants increased from 55 to 75˚C and then decreased with an increase in temperature to 85 and 95˚C.

Since at 75˚C, COD reduction is highest, therefore at 75˚C; effect of catalyst mass loading in the range of 2–4 kg/m^3 was studied. Values of k of Eq. (4) were found to be 0.00125, 0.001634, and 0.0022 dm$^{1.5}$/(mg$^{0.5}$ h), respectively, at catalyst mass loading 2, 3, and 4 kg/m^3 (Fig. 7(a)–(c)). The values of k were found to increase with catalyst mass loading from 2 to 4 kg/m^3. At 75˚C and catalyst mass loading 2–4 kg/m^3, order with respect to catalyst mass loading was evaluated by plotting Eq. (5) as ln k values at 2, 3, and 4 kg/m^3 vs. corresponding values of ln C_{w} (Fig. 7(d)). Order with respect to catalyst mass loading at these operating conditions was found to be 0.805.

Thermolysis of distillery effluents has been carried out by some investigators. Belkacemi et al. [15] found the degradation rate to be first order with respect to TOC during thermolysis of timothy grass-based distillery spent wash in high-temperature and high-pressure process. Lele et al. [16] found the degradation rate to be zero order with respect to COD during thermolysis of molasses-based distillery effluent.

3.7. Filtration

Filtration is one of the processes used to separate liquid and solid from slurry. To find out the filtration parameter, gravity filtration of the slurry was carried out at room temperature with Wattman filter paper (No. 42) supported on a Buchner funnel, under constant pressure filtration. The filtrate volume obtained as a function of time was observed. The filtration resistances for the filter media as well as the filter cake were obtained using the filtration equation [17].

$$\frac{\Delta t}{\Delta V} = \frac{\mu \alpha C}{A^2 \Delta P} \cdot V + \frac{\mu}{A \Delta P} \cdot R_{\mathrm{m}} \tag{6}$$

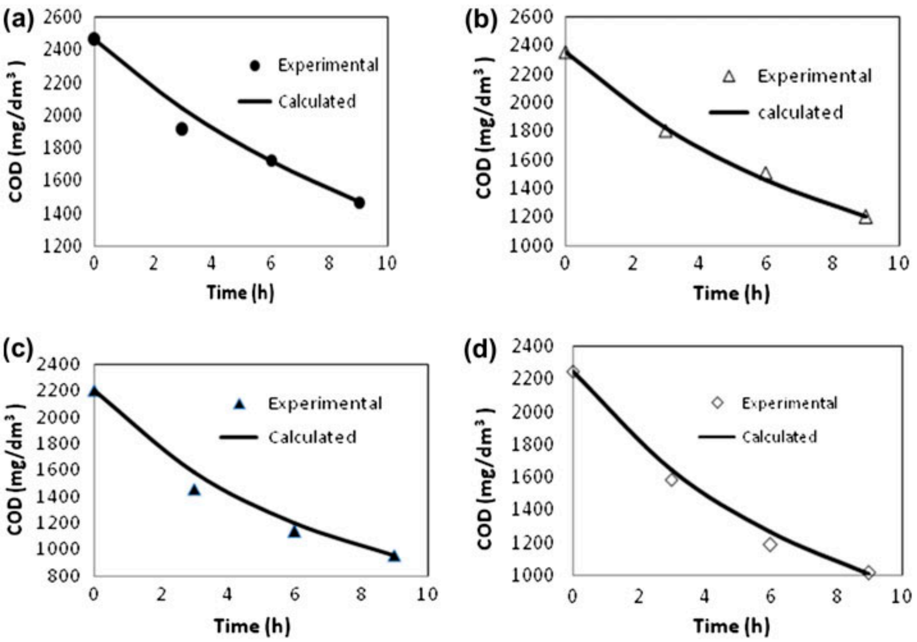

Fig. 6. Second-order kinetics plot of thermolysis process using CuO catalyst C_w = 4 kg/m^3. (a) 55°C. (b) 65°C. (c) 75°C. (d) 85°C.

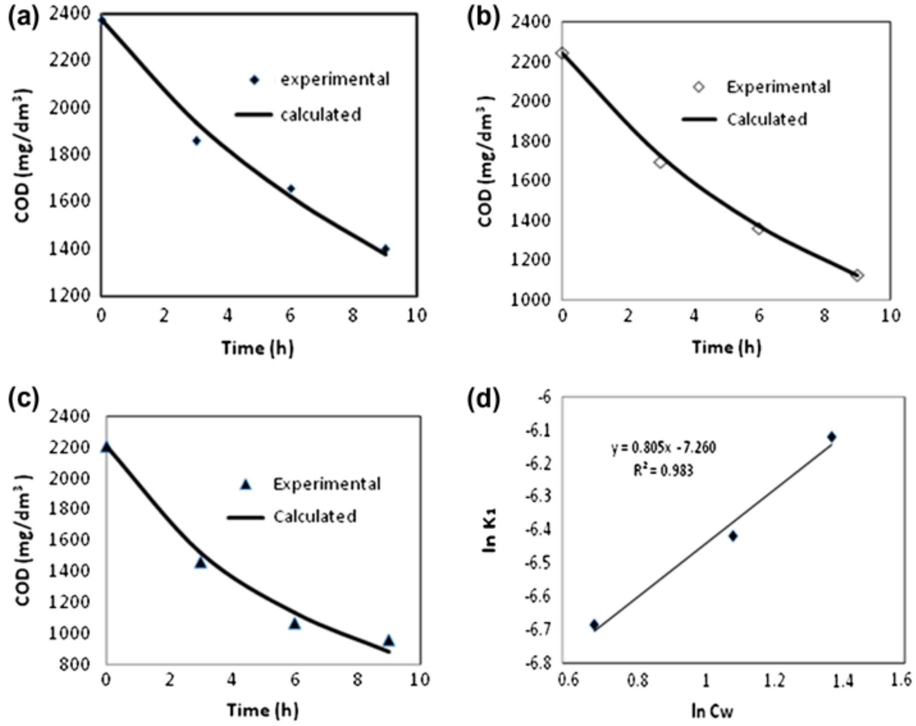

Fig. 7. Second-order kinetics plot of thermolysis process using CuO catalyst at pH 10 and temperature 75°C. (a) C_w 2 kg/m^3. (b) C_w 3 kg/m^3. (c) C_w 4 kg/m^3. (d) Plot for rate constant and catalyst mass loading.

where Δt is the time interval of filtration (s), ΔV is the filtrate volume collected up to that time interval (m^3), C is the solids concentration in the slurry (kg/m^3), V is the total liquid filtrate volume collected up to the time interval t (m^3), μ is the viscosity of the liquid filtrate (Pa s), ΔP is the pressure drop across the filter is equal to ρgh (Pa), A is the filtration area (m^2), R_m is the resistance of the filter medium (m^{-1}), and α is the specific resistance to filtration, also called as SCR.

After recording the volume of the filtrate as a function of time, a plot of $\Delta t/\Delta V$ vs. V produced a straight line, which is shown in Fig. 8. The values of R_m and α were evaluated from the slope and the intercept of plot and are presented in Table 2. Values of filter media resistance were found to be 1.43×10^{11}, 1.53×10^{11}, 1.37×10^{11}, 1.71×10^{11}, and $1.72 \times 10^{11}\ \mathrm{m}^{-1}$, and specific cake resistance was 15.01×10^{13}, 13.2×10^{13}, 10.08×10^{13}, 10.2×10^{13}, and 12.19×10^{13} m/kg at 55, 65, 75, 85, and 95˚C, respectively. Filter media resistance is important at the initial stage of filtration only. During filtration, first drop of filtrate appeared for the treatment at 75˚C as compared to the treatment at other temperatures. The values of specific cake resistance have been reported in the range of $4–12 \times 10^{13}$ m/kg for activated sludge and $3–30 \times 10^{13}$ m/kg for digested sludge [18]. Filtration data have also been evaluated previously for pulp and paper mill and textile mill wastewater treatment [6,8]. Analysis of residues is presented in Table 3.

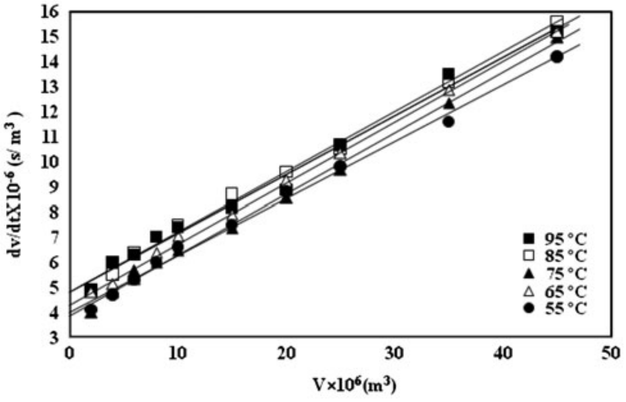

Fig. 8. Filtration studies for thermally treated SIWW.

3.8. Settling

The settling characteristic of the slurry mixture of treated effluent at temperatures 55–95˚C and pH 10 using CuO catalyst was studied. The process was performed to see the effect of the temperature on the settling characteristic of the precipitate. The treated SIWW sample after thermal treatment was slowly mixed and placed in 100 ml cylinder. Settling rate was found in order at 75˚C > 65˚C > 85˚C > 95˚C > 55˚C (Fig. 9). The variation in the settling rate of flocks with temperature may be probably due to their size and compactness. The physical property of residues varied with temperature. Eighty-five percent settling was observed in 90 min at 75˚C, which is considered as very good settling characteristics. Thus, it is easy to separate liquid and sludge.

In sedimentation, it was observed that during initial period of sedimentation, rate is slow, which is due to Brownian motion of particles. After this, rate of sedimentation increased linearly, showing constant settling zone. At the end of this zone, compression zone begins, at which liquid–solid intercepts became almost constant. Although a number of methods are available to calculate a compression zone to design continuous thickeners from the batch sedimentation data [19–22], the method proposed by Richardson et al. [19] is still preferred to design a continuous thickener based on the single batch sedimentation test. It gives a high precious value of changing nature of the flocs and their settling and compression characteristics.

Table 2
Filterability of the treated wastewater

S. No.	Initial pH (˚C)	Kp × 10^{-12}s/m^6	$\beta \times 10^{-6}$s/m^3	Cg/dm^3	$\alpha \times 10^{-13}$ m/kg	$R_\mathrm{m} \times 10^{-11}$ m^1
1	95	0.228	4.821	3.22	12.19	1.72
2	85	0.240	4.788	3.45	10.20	1.71
3	75	0.244	3.842	3.58	10.08	1.37
4	65	0.231	4.273	2.58	13.2	1.53
5	55	0.244	4.006	2.21	15.01	1.43

Table 3
Analysis of residue obtained after thermolysis at different temperature

S. No.	Parameters	95°C	85°C	75°C	65°C	55°C
1	Weight of residue (kg/m^3)	3.22	3.45	3.58	2.58	2.21
2	Color	Dark brown	Brown	Brown	Brown	Brown
3	Nature	Easy to grind	Easy to grind	Easy to grind	Easy to grind	Easy to grind
4	Size (mm)	0.5–3	0.5–3	0.5–3	0.5–3	0.5–3
5	Approximated drying period (h)	5	5	6	7	7
6	Percentage convertible COD	69.8	72.5	74	67.3	60
7	Percentage color reduction	74	77	80	52.6	65

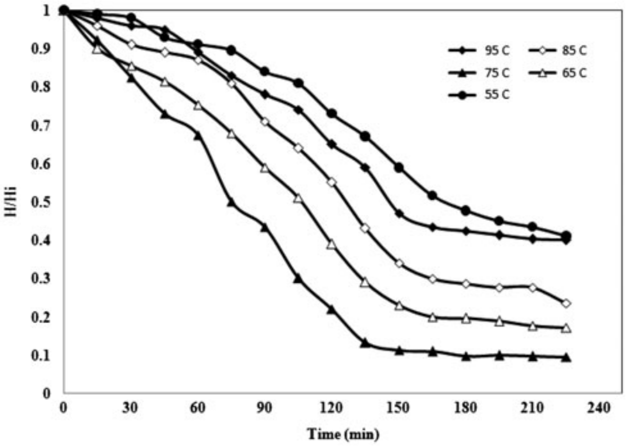

Fig. 9. Settling characteristics of thermally treated SIWW.

4. Conclusion

The thermolysis process proved to be an effective process to treat SIWW. The performance of catalyst was found to depend on pH and temperature. Copper oxide was found to be the best catalyst giving 74% COD reduction and 80% color reduction with a catalyst concentration of 4 kg/m^3, pH 10, and 75°C. COD reduction was accompanied by the formation of a settleable solid residue. Rate equation was found to 1.5 order with respect to COD and 0.805 order with respect to catalyst mass loading. Filtration and settling characteristics were found to be temperature dependent. The final pH of the treated effluent slightly decreased below its initial pH. The organic load of treated effluent can be reduced further by wet oxidation or membrane separation to reach zero discharge level.

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