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Treatment of ink production wastewater by chemical precipitation coupled with *Cyperus alternifolius*: pigments, organic compounds and ammonium removal

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ABSTRACT

Ink production wastewater is difficult to treat due to it consisted of pigments, acrylic resin, monoethanolamine, NH_4OH and additives, which have the effect of increasing of chemical oxygen demand (COD), ammonium nitrogen (NH_4^+–N), and suspended solids (SS). Therefore, the potential of chemical treatment of this wastewater coupled with phytoremediation was investigated. When treated with sulfuric acid, COD was decreased from 28,827 to 1,354 mg L^{-1}, and SS from 2,917 to 32 mg L^{-1}, while NH_4^+–N decreased from 458 to 295 mg L^{-1}. Thereafter, precipitation by MgO + H_3PO_4 was the optimum for ammonium precipitation, it could reduce ammonium to 98 mg L^{-1}. Adjusting system pH by $Ca(OH)_2$ for plant growth was better than using NaOH or KOH, due to the fact that calcium ions (Ca^{2+}) act as a nutrient. Phytoremediation by *Cyperus alternifolius* was investigated in the final treatment. It could decrease COD and NH_4^+–N to 329 and 13 mg L^{-1}, respectively, and passed the effluent standards.

Keywords: Ink production wastewater; *Cyperus alternifolius*; Precipitation; Phytoremediation

1. Introduction

Flexographic printing uses water–based inks to produce packaging materials. Water–based inks have been widely used in printing the packaging for foods, toys, wine, etc. The main pollutants in wastewater are pigments, acrylic resin, monoethanolamine (MEA), and NH_4OH. They cause wastewater to have alkaline pH and high levels of chemical oxygen demand, ammonium nitrogen and suspended solids. The wastewater generated from this process is highly colored and has a high–odor smell. The quality and quantity of wastewaters varies with the different processes of ink production, such as the types of inks, pigments and additives used. This wastewater cannot be discharged directly into the environment without treatment due to its deleterious effects on human health and the environment. Moreover, it also has aesthetical effects, even if the color is discharged at low concentrations, especially wastewater from ink production factories.

At present, the demand for ink will increase with the progress of the economy, resulting in increasing wastewater discharge from industry. The biological treatment of pigment in wastewater is effective in decolorization, but in some case it fails due to the presence of high metal and non–biodegradable organics in the wastewater [1,2]. Therefore, physicochemical methods are employed in the treatment. Many different techniques of physicochemical methods have been reported for treatment of ink production wastewater, such as Fenton process and ozonation [3], electrochemical treatment [4,5], nanostructured photocatalysts [6], and coagulation/flocculation process using biopolymers [7]. Although, these methods could reduce the color and turbidity of wastewater, COD

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and ammonium concentrations were still at high values. It was found that COD and ammonium were removed at a slower rate. To find an efficient way to remove nitrogen, the method of chemical precipitation has been studied widely for treatment of high ammonium nitrogen wastewater such as adding magnesium salt and phosphate to form magnesium ammonium phosphate hexahydrate [8,9]. In addition, constructed wetlands are designed to remove nitrogen and organic matter in wastewater. It is the conventionally used strategies for nutrient removal [10,11].

Therefore, this research study was combined chemical precipitation and phytoremediation in order to obtain the optimum condition for treatment of ink production wastewater. Phytoremediation was applied for cleanup of this wastewater at the final step. From our study, *Echinodorus cordifolius* [12], *Cyperus alternifolius* [13], *Thalia geniculate* [14], *Acorus calamus* [15], and *Dracaena sanderiana* [16] were used for screening the best plant for COD removal. It was found that *C. alternifolius* (66%) had the highest efficiency for COD removal followed by *E. cordifolius* (65%), *A. calamus* (57%), *T. geniculate* (56%), *D. sanderiana* (30%), respectively. Moreover, *C. alternifolius* could endure to toxicity of wastewater while *E. cordifolius* could not survive. Therefore, *C. alternifolius* was selected to study in this research. With this study, the combination method of chemical and phytoremediation were realized for completely color, COD, and ammonium removal, a new simplified method was proposed in our study.

2. Materials and methods

2.1. Pretreatment of wastewater with sulfuric acid and ferric sulfate

Original wastewater was obtained from a factory that produced pigments and ink for flexography printing of packaging (Thailand). The wastewater was of dark color, had a strong odor and showed high COD and high ammonium concentration as shown in Table 1.

There are many coagulants reported for wastewater precipitation; we decided to use inexpensive chemicals and an easier method. Experiments were carried out in glass pot of 10 L capacity. Chemical precipitation for removal of pigments from the wastewater used sulfuric acid (H_2SO_4) and ferric sulfate ($Fe_2(SO_4)_3$) [17,18]. The appropriate concentration of H_2SO_4 was added directly to the original wastewater in the range of 0.05%–0.2% (v/v) and $Fe_2(SO_4)_3$ was added in the range of 2%–3% (w/v) and then the solution was stirred for about 1 min for flocculation. After precipitation, the supernatant was analyzed for COD, pH, total Kjeldahl nitrogen (TKN), NH_4^+–N, and SS. The best method for precipitation of ammonium with various forms of magnesium phosphate was investigated.

2.2. Types of magnesium phosphate precipitate

Experiments for ammonium precipitation were performed with various chemicals such as (i) $MgCl_2\cdot6H_2O + Na_2HPO_4\cdot12H_2O$, (ii) $MgO + H_3PO_4$, and (iii) $MgSO_4\cdot7H_2O + Ca(H_2PO_4)_2$. First, the reagents were added to the wastewater (1 L) at $Mg^{2+}/NH_4^+/PO_4^{3-}$ molar ratios of 1:1:1 [19]. Second, the reaction solution was agitated by magnetic stirrer for 15 min and pH adjusted for precipitation with various alkaline chemicals (NaOH, $Ca(OH)_2$, and KOH) at pH 8.5–9.0. Third, the precipitate formed was allowed to settle out of the reaction solution and the supernatant collected for phytoremediation study.

2.3. Phytoremediation method

C. alternifolius, about 3–4 months old, was obtained from a garden market. Plants were washed carefully with tap water to remove soil and any contaminated substances from their roots. After that, they were pre–cultured in tap water for 5 d and the healthy plants were selected. Plants of similar sizes were used in the experiment. Clay soil for this study was obtained from a garden market. Soil samples were air–dried and ground, then sieved through a 0.47 cm sieve and mixed.

The experiments were performed in glass containers, it has 17 cm of diameter and 28 cm of height. Under plant grown in soil–containing pretreated wastewater, 300 g of plants and soil were used while under soil–containing pretreated wastewater, 300 g of soil was added in each pot. After that, 2 L of pretreated wastewater was added in the

Table 1
Characteristics of ink production wastewater before and after treatment by sulfuric acid and ferric sulfate

Parameter	Wastewater	Wastewater + H_2SO_4	Wastewater+ $Fe_2(SO_4)_3$	Effluent standard value[a]
Appearance				Not objectionable
COD (mg L^{-1})	28,827 ± 385	1,354 ± 49	1,287 ± 31	120–400
pH	7.45 ± 0.01	1.87 ± 0	2.29 ± 0.2	5.5–9
TKN (mg L^{-1})	514 ± 32	369 ± 27	387 ± 16	100–200
NH_4^+–N (mg L^{-1})	458 ± 15	295 ± 23	291 ± 21	–
SS (mg L^{-1})	2917 ± 34	32 ± 6	62 ± 8	50–150

[a]Industrial Effluent Standard Notification from the Ministry the Ministry of Science, Technology and Environment, 1996, Thailand

pot experiments. The experiments were operated effectively without the addition of an external carbon source during the experiment period of 3 d. The experimental work of phytoremediation was conducted in a green house in the outdoor conditions with natural light at 30°C ± 2°C, 68% ± 5% humidity. All experiments were performed in three replicates in each treatment.

2.4. Sampling and analytical methods

All samples were filtered using Whatman GF/C glass microfiber filter paper to remove solids. Concentrations of TKN, COD, NH_4^+–N, and NO_2^-–N were measured according to the standard methods [20].

TKN was measured by the digestion-titration method using an analyzer (kjeldatherm, Gerhardt, Germany). NH_4^+–N was measured by the titration method using the Rapid Distillation System (vapodesk 20, Gerhardt, Germany). COD was measured by the reactor digestion method using COD digestion vials (Odyssey DR/2500, Hach, USA), NO_2^-–N by the diazotization method (Odyssey DR/2500, Hach, USA), and NO_3^-–N by the cadmium reduction method using NitraVer 5 reagent (Odyssey DR/2500, Hach, USA). The procedure was followed according to the Hach DR/2500 spectrophotometer manual.

2.5. Statistical analysis

The data were analyzed statistically using the SPSS statistics software to perform a one–way analysis of variance (ANOVA). Significantly different means were assessed by a Duncan's multiple–range test ($p < 0.05$).

3. Results and discussion

3.1. Comparison of wastewater treatment by sulfuric acid and ferric sulfate

In this study, treatment of original wastewater using sulfuric acid and ferric sulfate as precipitants was compared for the efficiency of pigment and SS removal. This wastewater is weakly alkaline and negative charged (–45 mV), which might be due to come from hydroxide ions or negatively charged colloidal particles and led to coagulative aggregation. Since coagulant is required for precipitation of hydroxyl group or negatively charged compounds, which neutralized by cations from iron and hydrogen ions (sulfuric acid) [17,18]. Both $Fe_2(SO_4)_3$ and H_2SO_4 had affected on physical and chemical of colloidal particles changes including, charge neutralization, destroy the micelle stability, thus forming flocculation and then precipitation.

After original wastewater precipitation, the results revealed that both precipitants could precipitate wastewater well. The results showed that H_2SO_4 had a lower efficiency in COD removal than $Fe_2(SO_4)_3$ but it had a high efficiency to remove pigments and SS, as shown in Table 1. Although, ferric sulfate could provide a large number of complex ion and strong adsorption of colloid particles, using $Fe_2(SO_4)_3$ as a precipitant caused the appearance of an iron color in the treated wastewater (see Table 1). For this reason, H_2SO_4 was chosen for treatment. Moreover, the color was reduced from a dark color to light orange (Table 1).

3.2. Removal of ammonium nitrogen removal by various types of magnesium phosphate

Wastewater after pretreatment with sulfuric acid still had a high ammonium concentration, which could affect eutrophication or algal bloom (Table 1). Plants can absorb and use ammonium as a nitrogen source, but it takes too long a time. Therefore, wastewater precipitation with magnesium phosphate was studied for ammonium removal in a shorter time such as $MgCl_2{\cdot}6H_2O + Na_2HPO_4{\cdot}12H_2O$, $MgO + H_3PO_4$, and $MgSO_4{\cdot}7H_2O + Ca(H_2PO_4)_2$. This technique can remove ammonium by forming magnesium ammonium phosphate precipitate which has been studied for different types of wastewater such as municipal and industrial wastewater [21,22], anaerobically pretreated domestic wastewater [23] and landfill leachate [24]. The cost of precipitation process is roughly similar to the cost of nitrification and denitrification of communal wastewater [25]. In experiment, the results showed that precipitation of original wastewater with $MgCl_2{\cdot}6H2O + Na_2HPO_4{\cdot}12H_2O$ could decrease ammonium better than $MgO + H_3PO_4$ or $MgSO_4{\cdot}7H_2O + Ca(H_2PO_4)_2{\cdot}H_2O$. But the wastewater still appeared dark–colored (Table 2), it was not precipitated. However, precipitation of wastewater + H_2SO_4 by magnesium phosphate had a higher efficiency of ammonium removal and wastewater was precipitated. The results indicated that $MgCl_2{\cdot}6H_2O$ + $Na_2HPO_4{\cdot}12H_2O$ could remove ammonium to approximately 61 mg L^{-1} from the initial 458 mg L^{-1}. MgO + H_3PO_4 and $MgSO_4{\cdot}7H_2O$ + $Ca(H_2PO_4)_2{\cdot}H_2O$ could remove ammonium to 98 and 201 mg L^{-1}, respectively.

However, the byproduct sodium chloride (NaCl) was obtained when precipitating wastewater with $MgCl_2{\cdot}6H_2O + Na_2HPO_4{\cdot}12H_2O$ (Eq. 1) affected plant survival. Therefore, the combination of MgO + H_3PO_4 represented the best conditions for ammonium removal.

$$MgCl_2{\cdot}6H_2O + Na_2HPO_4{\cdot}12H_2O + NH_4^+ \rightarrow MgNH_4PO_4{\cdot}6H_2O\downarrow + 2NaCl + 12\,H_2O + H^+ \quad (1)$$

$$MgO + H_3PO_4 + NH_4^+ + 6H_2O \rightarrow MgNH_4PO_4{\cdot}6H_2O\downarrow + H_2O + H^+ \quad (2)$$

$$MgSO_4{\cdot}7H_2O + Ca\left(H_2PO_4\right)_2{\cdot}H_2O + NH_4^+ \rightarrow MgNH_4PO_4{\cdot}6H_2O\downarrow + CaSO_4\downarrow + PO_4^{3-} + 2H_2O \quad (3)$$

3.3. Phytoremediation studies on ink production wastewater by C. alternifolius

The system pH for ammonium precipitation with MgO + H_3PO_4 was 8.5–9.0. Therefore, NaOH, KOH and $Ca(OH)_2$ were used to adjust system pH. After treatment for 3 d, the remaining concentrations of ammonium under plants grown in soil–containing wastewater conditions were 8, 17, and 13 mg L^{-1} by adjusting pH with NaOH, KOH and $Ca(OH)_2$, respectively (Table 3). *C. alternifolius* could live and tolerate in ink production wastewater. Moreover, it had a high stomata conductance that related to a high of gas exchange and water

Table 2
Comparison of three different chemicals to precipitate original wastewater and wastewater + H_2SO_4

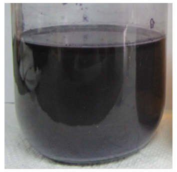
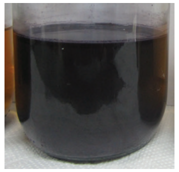
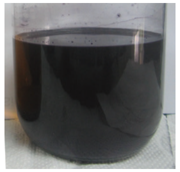
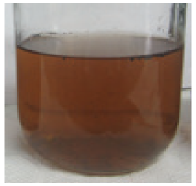
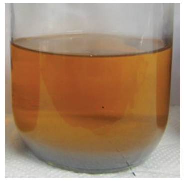
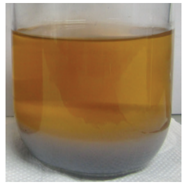
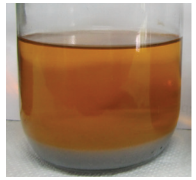
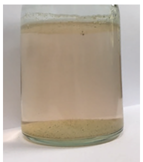
Parameter	Precipitation with chemical substances							
	Original wastewater				Wastewater + H_2SO_4			
		$MgO + H_3PO_4$	$MgSO_4.7H_2O$ + $Ca(H_2PO_4)_2.H_2O$	$MgCl_2.6H2O$ + $Na_2HPO_4.12H_2O$		$MgO + H_3PO_4$	$MgSO_4.7H_2O$ + $Ca(H_2PO_4)_2.H_2O$	$MgCl_2.6H2O$ + $Na_2HPO_4.12H_2O$
Appearance								
NH_4^+–N (mg L^{-1})	458 ± 15	309 ± 9	380 ± 13	160 ± 10	295 ± 23	98 ± 8	201±10	61±7

Table 3
Effect of NaOH, KOH, and $Ca(OH)_2$ on wastewater + H_2SO_4 + MgO + H_3PO_4 treatment by *C. alternifolius* for 3 days

Parameter	Wastewater			Wastewater + Soil		Wastewater + Soil + Plant		Appearance of plant after 3 days
	Day 0	Day 1	Day 3	Day 1	Day 3	Day 1	Day 3	
(a) NaOH								
COD (mg L^{-1})	1,564 ± 55	1,463 ± 32	1,252 ± 49	1,117 ± 98	771 ± 27	862 ± 24	554 ± 24	
pH	8.81 ± 0.2	8.71 ± 0.01	8.48 ± 0.04	8.15 ± 0.07	8.15 ± 0.02	7.75 ± 0.02	7.83 ± 0.02	
TKN (mg L^{-1})	57 ± 6	39 ± 7	21 ± 3	33 ± 6	9 ± 2	20 ± 6	20 ± 2	
NH_4^+–N (mg L^{-1})	35 ± 4	13 ± 1	11 ± 1	8 ± 2	4 ± 1	2 ± 1	19 ± 1	
(b) KOH								
COD (mg L^{-1})	1,320 ± 12	1,273 ± 5	1,092 ± 5	1,056 ± 13	687 ± 2	781 ± 4	495 ± 8	
pH	8.50 ± 0.03	8.37 ± 0.02	8.37 ± 0.02	7.97 ± 0.02	7.92 ± 0.01	7.03 ± 0.02	7.09 ± 0.01	
TKN (mg L^{-1})	44 ± 8	56 ± 9	24 ± 7	29 ± 6	18 ± 5	21 ± 6	20 ± 7	
NH_4^+–N (mg L^{-1})	29 ± 9	29 ± 4.04	20 ± 2	22 ± 2	16 ± 1	17 ± 3	17 ± 2	
(c) $Ca(OH)_2$								
COD (mg L^{-1})	1,488 ± 15	1,283 ± 48	950 ± 26	977 ± 33	632 ± 25	736 ± 32	329 ± 37	
pH	8.74 ± 0.01	8.59 ± 0.12	8.44 ± 0.02	7.92 ± 0.02	7.92 ± 0.02	7.04 ± 0.06	7.04 ± 0.01	
TKN (mg L^{-1})	49 ± 7	47,111	44 ± 13	37 ± 7	4119	30 ± 8	24 ± 7	
NH_4^+–N (mg L^{-1})	32 ± 7	30 ± 11	32 ± 1	18 ± 1	20 ± 1	14 ± 2	13 ± 2	

Table 4
Characteristics of ink production wastewater after treatment by combination three methods

Parameter	Wastewater	Wastewater + H_2SO_4 + (MgO + H_3PO_4) + $Ca(OH)_2$	Wastewater + $Fe_2(SO_4)_3$ + (MgO + H_3PO_4) + $Ca(OH)_2$	Effluent standard value[a]
Appearance				Not objectionable
COD (mg L^{-1})	20,077 ± 87	1,216 ± 25	1,041 ± 31	120–400
pH	7.28 ± 0.02	8.53 ± 0.02	8.60 ± 0.2	5.5–9
TKN (mg L^{-1})	486 ± 4	74 ± 4	84 ± 1	100–200
NH_4^+–N (mg L^{-1})	331 ± 2	65 ± 2	79 ± 4	–
SS (mg L^{-1})	2,125 ± 21	27 ± 3	35 ± 3	50–150

[a]Industrial Effluent Standard Notification from the Ministry the Ministry of Science, Technology and Environment, 1996, Thailand

loss from transpiration [26]. These properties of plant had a potential application in phytoremediation.

However, NaOH and KOH had an effect on plants, as the plant leaves yellowed and wilted, followed by plant stems beginning to wither and dry. High concentration of Na^+ resulted in ionic stress, to which the plants could not tolerate and adapt. An increase in Na^+ content during treatment is thought to result from the competition between the transport and accumulation of Na^+, leading to osmotic shock in roots and to molecular damage, inhibiting plant growth and then death. This is the result of the inhibition of many enzymes. Therefore, it is important to prevent Na^+ accumulating to a high level in the cytoplasm or in organelles other than the vacuoles [27]. Meanwhile, the excess potassium is not toxic to plants, but it limits the efficient absorption of other minerals and nutrients leading to lots of other deficiency such as magnesium, manganese, zinc and iron. This in turn results in poorly developed plants with a yellowish color and stunted growth due to a molecular imbalance [28].

When plants were exposed to $Ca(OH)_2$, plant leaves and plant stems were still healthy and new roots and shoots appeared. It is possible that $Ca(OH)_2$ did not affect the plants and was suitable for plant growth. Moreover, adjusting system pH with $Ca(OH)_2$ had the highest efficiency in COD removal (Table 3(c)). This might be due to the fact that the plants could tolerate exposure to Ca^{2+} and could adapt and use $Ca(OH)_2$ as a nutrient. Calcium ions (Ca^{2+}) act as an osmoticum within vacuoles, a stabilizing element of membranes, a strengthening agent in cell walls, and a secondary messenger for a multitude of signals [29,30]. In addition, plants could also adsorb color, from an orange color to clear within 3 d.

3.4. Combination method for ink production wastewater treatment

In this experiment, three methods of wastewater treatment as wastewater precipitation, ammonium precipitation, and alkaline addition for adjusting pH system was combined at the same time for experiment facility. Two experiments, precipitation wastewater with H_2SO_4 + (MgO + H_3PO_4) + $Ca(OH)_2$ and $Fe_2(SO_4)_3$ + (MgO + H_3PO_4) + $Ca(OH)_2$ were investigated. Original wastewater was added with H_2SO_4 or $Fe_2(SO_4)_3$ under stirring condition. After wastewater was occurred flocculation, MgO + H_3PO_4 was added and then followed by $Ca(OH)_2$ addition. The resulted showed that combination of three methods could reduce SS, COD, and ammonium contents (Table 4). Interesting that combination method with $Fe_2(SO_4)_3$ + (MgO + H_3PO_4) + $Ca(OH)_2$ could remove iron color. However, it showed strong iron color in sediment waste. Therefore, combination of H_2SO_4 + (MgO + H_3PO_4) + $Ca(OH)_2$ could be apply in ink production wastewater treatment before phytoremediation method.

4. Conclusions

Treatment of ink production wastewater by sulfuric acid and then precipitation by MgO + H_3PO_4, coupled with *C. alternifolius* could decrease color, COD, ammonium and SS. The results revealed that MgO + H_3PO_4 showed efficiency in ammonium removal, and NaCl did not occur in the products to affect the plants. Moreover, $Ca(OH)_2$ was the best alkali for adjusting pH because $Ca(OH)_2$ is a nutrient for plant growth. This condition could decrease ammonium and COD to 13 and 329 mg L^{-1}, respectively, and color could be removed to clear within 3 d.

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