Performance Assessment of Recycled Alum Sludge in the Treatment of Textile Effluent Containing Dye

Mbodi TN*
Department of Civil Engineering, Cape Peninsula University of Technology, Cape Town, South Africa

Abstract
Textile industry is considered as one of the most polluting sectors in terms of discharge and effluent composition, such as dye, which represents an environmental hazard when discharged without any proper treatment. A study was conducted to investigate the capability of the use of recycled alum sludge (RAS) as an alternative treatment for the reduction of colour, chemical oxygen demand (COD), and pH adjustment from dye based synthetic textile industry wastewater. The coagulation/flocculation process was studied for coagulants of Alum-RAS ratio of 1:1, 1:2 and 0:1 and the results were evaluated against the removal efficiency of fresh alum in the treatment synthetic wastewater. Fresh alum recorded a colour removal efficiency of 89 ± 2%, while 78 ± 3%, 86 ± 3%, 82 ± 2% were recorded during the treatment with coagulants with mix ratios of 0:1, 1:1 and 1:2, respectively. Evidences have demonstrated that that reusing the alum sludge as a low-cost material pre-treatment method into the coagulation/flocculation process can offer some advantages such as high removal efficiency for disperse dye and economic savings on overall treatment of the industry wastewater.

Keywords: Alum; Coagulation/flocculation; Dye; Recycled alum sludge; Textile wastewater

Introduction
The textile industry refers to a collection of related industries that is involved with various manufacturing activities such as spinning, weaving, dyeing, printing, and finishing [1]. The various processes may be disseminated, but in some instances, one facility may be responsible for the entire production pipeline from raw material to finished product. Final products include clothing, knitwear.

There are many types of textiles that are produced using a variety of raw materials and manufacturing processes in order to obtain the final products such as garments, curtains, and carpets [2]. Consequently, the amount of water used and the quality of the wastewater generated by each process is highly variable. The variable water quality is characterised by parameters such as the specific water intake, specific effluent volume, and specific pollutant load [3]. In general, between 70% and 80% of water used by the textile industry is discharged as wastewater. This effluent typically contains high concentrations of pollutants such as dissolved solids, recalciitrant organic matter, heavy metals, and dyes, which can be detrimental to the environment if not adequately treated prior to discharge [4-7].

The current trend in South Africa is to ensure the water resources such rivers, dams and ground water are not polluted by effluent of poor quality. This has led to the development and implementation of various regulations such as the National Water Act [8]. The environmental regulations require that effluent containing harmful pollutants should be treated to comply with specified standards prior to discharge. Numbers of textile industries are not compliant with the regulation.

Chemical coagulation has often displayed high treatment efficiencies in the removal of dyestuff contained in dyehouse wastewater. However, this treatment method generates high volumes of coagulant sludge that requires a specialised handling and disposal to avoid pollution of the environment. With the implementation of new stricter regulation, disposal of sludge via methods such as land filling may be prohibited in future. Studies have shown that traces of coagulants found in the sludge after flocculation still possess adsorbing power and could be reused in the coagulation-flocculation process. This study evaluates the capabilities of recycled coagulant sludge in the treatment of dye house wastewater.

Experimental Work
Preparation of synthetic wastewater
Synthetic wastewater was produced by diluting Disperse Blue Dye 1 (C_18H_{13N_3O_6} (Sigma-Aldrich) in distilled water. A stock dye solution of 1000 mg/l was prepared in deionised water. The stock solution was then diluted to the working concentration of 12.5 mg/l.

Coagulation/flocculation process
The synthetic wastewater was treated using a Stuart Scientific Flocculator SW1 with six 1000 ml Pyrex beakers. Before the start of the mixing, aluminium sulphate (Al(SO_4)_{18}H_2O) was added to the wastewater at a coagulant at a concentration of 100 mg/l. The treatment process was conducted in a batch mode

Immediately after mixing, the liquor was mixed at an initial speed of 80 rpm for 1 min. The rotating speed was then reduced to 30 rpm and maintained for a period of 20 min. Thereafter the machine was stopped to allow the settling of particles in the wastewater for 60 min.

After settling for 60 min, the supernatant was collected and analysed. The settled wet sludge was collected and dewatered. The dry alum sludge was then recyled being used as a coagulant in conjunction with fresh alum at ratios (fresh alum: recycled alum sludge) of 1:1, 1:2 and 0:1. The effluent and the sludge produced were handled in the same manner as during the treatment with fresh alum.

*Corresponding author: Mbodi TN, Department of Civil Engineering, Cape Peninsula University of Technology, Cape Town, South Africa, Tel: 0614091954; E-mail: tmmbodi@hotmail.com, tmmbodi@gmail.com

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Results and Discussion

Effect of pH in coagulation/flocculation process

With the use of potassium hydroxide (KOH) that provided the alkaline conditions favourable for coagulation/flocculation by increasing the amount of hydroxide ions therefore increasing the formation of Al(OH), for higher removal of dye, experiment results have demonstrated that high removal efficiency of colour from synthetic wastewater can be achieved under alkaline conditions. This aligns with the findings from Huang et al. [9] who claims that the improvement of floc growth rate is more substantial at basic conditions which enhances the colour removal efficiency when alum is used in the treatment of wastewater containing dye. This claim was supported by findings from Chu [4], whom demonstrated an 85% of removal efficiency of colour with 100 mg/l of alum and a cationic polymer as a sludge thickener at a concentration of 32.5 mg/l and a pH of 9.1 in the treatment of synthetic wastewater containing disperse dye. Similar findings in the treatment of synthetic wastewater under alkaline conditions were also reported by Nair [10] and Huang et al. [9], with the former recording a colour removal efficiency of 89% when treating synthetic wastewater containing disperse dye with coagulant mix of alum and recycled alum sludge at a pH 9; and the latter removing 94% of colour from a synthetic wastewater containing reactive dye with alum a coagulant at a pH of 9. Bo et al. also supported the findings by stating in his research that the coagulation mechanism is transformed to enmeshment with increasing pH and larger flocs with more compact structures are formed in a shorter time [11]. The effectiveness of high pH level of the influent during the coagulation/flocculation process was also demonstrated in the treatment of the actual wastewater. Findings from Alisafi et al. have reported that 95% of colour was removed from the wastewater from a textile manufacturing plant after treatment by coagulation/flocculation with alum and NaCl at a pH level of 10; and Xu et al. reported that when treating tannery wastewater at a pH 8 with alum as a coagulant, a colour removal efficiency 90% can be achieved [12,13].

However, findings from Verma et al. contradicted the hypothesis by achieving high removal efficiencies during the treatment of synthetic wastewater containing disperse dye with alum in conjunction with catalysts and recording colour removal efficiencies of 79% at pH 5.3 and 74% at pH 4 after adding polyacrylamide-based polymer (PAM) and copper sulphate (CuSO₄), respectively, as catalyst [14].

These findings imply that high removal of disperse dye can be achieved during treatment by coagulation/flocculation with alum under alkaline conditions.

Performance analysis of coagulation/flocculation with recycled alum sludge effluent

Colour: According to the study, all coagulant ratios displayed substantial level of treatment in the removal of colour. The treatment with fresh alum (1:0) recorded the highest removal efficiency with an average of 89 ± 2% of colour removed during the treatment. This is due to the purity of the fresh alum particles, which allow a higher adsorption rate of pollutants in suspended and/or colloidal states from the body of water. The treatment with recycled alum sludge (0:1) observed the lowest average removal efficiency of 78 ± 3%. This loss in treatment capability was caused by the release of pollutants trapped in the sludge particles due to the reduction/saturation of surface areas of alum particles, therefore reducing the coagulant adsorptive power. By combining equal ratio of fresh alum and recycled alum sludge as a coagulant (1:1), an average colour removal of 86 ± 3% was recorded. It was observed that adding recycled alum sludge in the coagulant mix (1:2) would produce an average removal efficiency of 82 ± 2%. Despite having a minor impact on the colour removal efficiency, the increment of recycled alum sludge in the coagulant mix did not cause deterioration in the effluent quality. According to Chu, these findings were due to the added fresh alum that not only removes pollutant from the synthetic wastewater, but also adsorbs some of the pollutants released by saturated recycled alum sludge particles.

In this study, recycled alum sludge with and without added fresh alum was used for the removal of colour recorded treatment capabilities relatively as good as fresh alum. However, test results demonstrated that coagulant mix of fresh alum and recycled alum sludge (1:1) was highly efficient in the treatment of disperse dye from synthetic textile wastewater. This was supported by evidences from other studies that have also shown that coagulation/flocculation with alum and recycled alum sludge was effective for the removal colour. Furthermore, findings from this study suggested that the coagulant containing the ratio mix of 1:1 could provide a better alternative in the removal of colour from textile wastewater.

COD: The coagulation flocculation treatment with fresh alum/recycled alum sludge of mix ratio of 1:1 have displayed very poor treatment capabilities in the removal of COD from the synthetic textile wastewater. The highest treatment efficiency was recorded at 6%. However, increases of up to 14% in COD in the effluent after treatment were more often observed throughout the experiment. The increase in COD was mainly attributed to the excess of dissolved chemicals from the fresh coagulant in the wastewater, also to the dye particles released by the saturated particles of recycled alum sludge. The treatment with the coagulant of fresh alum/recycled alum sludge of mix ratio of 1:2 also recorded average treatment efficiency -9 ± 2%. The increase in COD is suspected to be mainly caused by the release of pollutants from the sludge. This is supported by the findings of Chu that claims that the excess saturated dye particles trapped in the sludge are restabilised when the coagulant has a ratio of recycled alum sludge in the sludge higher than the fresh alum. Yang and Verma also supported the claim by stating that the polluting particles that were trapped the sludge can be released back into aqueous phase during the treatment process. In the study conducted by Nair, it was stated when the amount of contaminant released by the sludge in the solution exceeds the removal by coagulation, the COD removal efficiency is reduced. This implies that the coagulation flocculation treatment with fresh alum/recycled alum sludge of mix ratio of 1:2 has very poor treatment capabilities in the removal of COD from the synthetic textile wastewater containing disperse dye.

Coagulation/flocculation treatment method recorded very poor treatment capabilities in the removal COD. Fresh alum recorded the highest efficiency with 29 ± 3%. This caused by fact that some of the coagulant particles remain dissolved in the effluent after treatment which results in an increase in the dissolved constituent in the wastewater. The treatment with recycled alum sludge recorded removal efficiency as low as 22 ± 3%. According to Chu, some of the polluting particles previously trapped in the recycled alum sludge will be released back to the aqueous phase and causing deterioration of the effluent quality since recycled alum sludge represents the only source that may result in effluent contamination. Effluents from the coagulant made of combined fresh alum and recycled alum sludge (1:1 and 1:2) have recorded little to no reduction of COD. In some case an increase of COD was recorded. This increase recorded was mainly caused by the excess of dissolved chemicals from the fresh coagulant and the
dye particles release by the saturated particles of recycled alum sludge. Based on the evidence, it was concluded recycled alum sludge (0:1) was the best alternative in the removal of COD from wastewater after fresh alum (1:0). In this study, recycled sludge with and without added fresh alum was used for the removal of COD recorded poor treatment capabilities. However, test results demonstrated that coagulant mix of fresh alum and recycled sludge (1:1 and 1:2) demonstrated very poor removal efficiency of COD in the treatment of synthetic textile wastewater containing disperse dye. This was supported by evidences from other studies that have also shown that coagulation/flocculation with alum and/or recycled sludge had very low treatment capabilities. Furthermore, findings from this study suggested that the coagulant containing the ratio mix of 0:1 provided the best alternative in the removal of colour from textile wastewater.

TDS: In this study, recycled sludge with and without added fresh alum was used for the treatment of TDS recorded limited treatment capabilities. However, test results demonstrated that coagulant mix of fresh alum and recycled sludge (1:1 and 1:2) demonstrated very poor removal efficiency of COD in the treatment of synthetic textile wastewater containing disperse dye. Additionally, findings from this study suggested that the coagulation/flocculant did not provide a good option in the removal of TDS from textile wastewater.

pH: The treatment with fresh alum recorded an important drop from a maximum of 12 in the raw wastewater down to a pH ranging between 8 and 9 in the treated effluent. This notable change in pH was attributed to the presence of alum in the wastewater that produced sulphuric acid in contact with water; as alum was the only chemical constituent added during the treatment process. The findings were supported by Verma who claimed that some of the particles of the alum used during the treatment process were dissolved in the wastewater and consequently causing a drop in pH level. However, the treatment with recycled alum sludge recorded very little change in pH, as it dropped from a maximum of 12.0 in the raw wastewater down to a pH range between 11.5 and 11.8 in the treated effluent. The little change in pH that occurred during the treatment is attributed to the limited amount of free alum particles to produce sulphuric acid in contact with water. According to Metcalf and Eddy, an excessive amount of coagulant may be required to lower the pH to the optimal pH ranges in water in high alkalinity [15]. An improvement in the change in pH was recorded during the treatment with fresh alum and recycled alum sludge (1:1), as a drop from a maximum of 12.0 in the raw synthetic wastewater down to a pH range between 9.8 and 10.6 in the treated effluent. These results were attributed to the addition of fresh alum to the coagulant. Evidences have shown that by adding fresh alum to recycled sludge a higher drop in pH, in comparison to 0:1, could be observed. According to Huang this change in pH that occurred during the treatment was due to the acidic nature of fresh sludge to produce sulphuric acid in contact with water and therefore reduce the pH of the treated effluent. Evidences have demonstrated that the treatment with coagulant mix ratio of 1:2 provided little change in the pH as a decrease from a range between 11.9 and 12.0 in the raw synthetic wastewater down to a pH ranging between 10.7 and 11.1 in the treated effluent was recorded. Findings from Chu and Huang suggested that although fresh alum promoted the drop in the pH level by producing sulphuric acid in contact with water, its effect was limited due to the important amount recycled sludge in the coagulant mix ratio which promoted a considerable release and/or restabilisation of dye particles back in aqueous form and therefore limiting the drop in pH.

Conclusion
In the case of this study, the treatment of synthetic wastewater with fresh alum has shown that high removal of colour and TDS can be achieved at pH surrounding 11.9. However, the treatment had low impact in the reduction COD from the wastewater. This implies that fresh alum is highly effective in the treatment of wastewater containing disperse dye. The treatment of synthetic wastewater with alum sludge as a coagulant has shown fair treatment capabilities in the removal of colour. However, the alum sludge has demonstrated poor removal efficiencies in the treatment of both COD and the TDS due to the release of particles previously trapped in the sludge. The limited amount of available free aluminium particle has resulted in a slight change in pH in the treated effluent. Therefore, recycled sludge as a coagulant can remove disperses dye from wastewater up to a certain extent. In the case of this study, the treatment of synthetic wastewater with a coagulant of fresh alum/recycled alum sludge mix ratio of 1:2 has shown that a high removal colour and a fair reduction of TDS can be achieved. However, the treatment has displayed very poor COD removal efficiencies. A pH reduction of up to 9.8 which suggests that the coagulant of fresh alum/recycled alum sludge mix ratio of 1:2 have high removal capabilities in the treatment of disperse dye from synthetic wastewater. According to evidences, the treatment of synthetic wastewater with fresh alum mixed with recycled sludge at a mix ratio of 1:2 as a coagulant has shown high colour removal efficiencies. However, the alum sludge has demonstrated poor removal efficiencies in the treatment of both the COD and the TDS due to the release of particles previously trapped in the sludge. The limited amount of available free aluminium particle has resulted in a slight change in pH in the treated effluent. Therefore, recycled sludge as a coagulant can remove disperses dye from wastewater up to a certain extent. Based on the evidence obtained from the comparative study conducted on the selected coagulant mix containing recycled sludge in terms of their capability to treat synthetic textile wastewater, coagulant containing fresh alum and recycled sludge at a mix ratio of 1:1 is found to offer the best alternative to fresh alum as it displayed an overall higher removal efficiency in the treatment of colour, COD and TDS from the synthetic wastewater.

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