

Utilization of Poly Inorganic Coagulants Impregnated with Activated Silica Derived from Rice Husk Ash in Treatment of Grey Water

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Abstract: The sanitation in Egypt, especially rural areas that have been a critical irritant should be faced. In the near future, this problem is predicted to expand with the increase of population and excessive use of drinking water. Accordingly, the research group decided to adopt the problem of sanitation in village regions to find the appropriate scientific solution from an environmental and economic point of view.

The work aims to treat of the grey water through removing suspended matters (TSS) and degradable and non-degradable organic materials (COD & BOD) by two stages, 1st stage; utilization coagulants such as, poly aluminum chloride silicate (PACSi), poly aluminum hydroxy sulphate silicate (PAHSSi), poly ferric chloride silicate (PFeClSi), and poly aluminum ferric chloride silicate (PAIFeClSi) through coagulation/flocculation/sedimentation and filtration steps. From the results of a grey water treatment using PAIFeClSi, the maximum removals of TSS, COD and BOD are 94.2, 89.2, and 76.9% respectively. PAIFeClSi is the superior poly inorganic coagulant copolymer may be assigned to the co-polymers of Si⁴⁺, Al³⁺ and Fe³⁺. Therefore, the developed rice husk ash poly inorganic co-agulants (PAIFeClSi) is considered as a better replacement technology for grey water treatment due to low cost and high efficacy in this application.

2nd stage: column system through anthracite bed, with adsorption capacity ($C_t/C_o = 50\%$ or $Q_{0.5}$) was 16.625 mg the column recycled at least five times and only was 5 ml of HCl (1M) for each regeneration and no loss in sorbent mass per cycle should be considered.

Keywords: Grey water; Poly Aluminum Ferric Chloride Silicate; Anthracite; Adsorption.

1 Introduction

Water administration is becoming progressively significant, essentially in water-rare sites of the world, such as, the Middle East and Gulf regions. In such districts, water fee is usually very high and, as such, there is a strong tendency to preserve and retrieve water. Further, the administration and treatment of sewage water are also becoming more and more remarkable due to charges correlated thereto, ecological interests, and stringent disposal standards.

Urban and rural wastewater administration has been a necessary for the birth of violently populated city and villages. Although it was originally applied for disease control through faeces removal from sewage water, modern urban and rural wastewater management has constantly changed throughout the 20th century in order to face new social requirements [1].

Today, wastewater management faces emerging concerns such as environmental pollution, resource recovery and the revival of greenhouse gases. Viable access for remedying this defiance is that of source separation systems for urban and rural sanitations. These systems, as proposed by Otterpohl et al., 2003 [2] separate domestic wastewater from storm water and fractionate the domestic wastewater into separate streams for more efficient treatment. Integrated source isolation connotation often concentrates on the isolation of toilet waste water (black water) with the possible addition of food waste and other domestic wastewater (grey water). By using source separated streams the potential for water reuse and recovery of energy and nutrients from wastewater can be increased. [3-5].

In regions where a domestic sewage system is not obtainable, decentralized sewage systems such as, for example, septic tanks can be used. The septic tanks usually have two chambers, with the 1st chamber extruding sewage water, and the 2nd chamber generating treated water to a drain field, which can spread over a large district, for example, from 200 to 300 m² for a three-bedroom house. The suspended solids in the sewage water fall to the base of the first chamber while froth stays on the surface. A barrier between the first and second chambers has an orifice that permits scummy water to flow from the 1st to the 2nd chamber where the extra settlement of suspended solids in the water can happen. Anaerobic bacterial activity without the existence of oxygen in the first and second chambers turns the suspended solid precipitates into sludge. The liquid present in the 2nd chamber directly exits the septic tank, into the drain field where the residual impurities present in the water decay in the soil [6]. Urine separation from source alludes to the separate collection of human pee and excreta at the point of their production, i.e. at the toilet or urinal. Separation of pee from excreta allows human waste to be managed individually and utilized as a prospective resource [7].

The cause for preserving human pee and excreta separately in a dry latrine rather than in a pit latrine is to minimize odor as mixing of human pee and excreta causes intrinsic odor, eschew production of moist, odorous fecal sludge, which has to be eliminated by someone when the pit latrine is full, enables fast drying of feces which makes handling of feces more simple and hygienic, reduce environmental impacts and allow for the recovery of urine, which, can be reused as fertilizer [8]. The justification of separate treatment of the two types of human pee and excreta is nearly disinfected and low in micro-organism, as well as isolation is healthful. This means that pee after incubation can be easily utilized as a manure and plant food or discarded with minimal hazard to society [9].

The grey water rich in organic materials with a high content of biological oxygen demand (BOD), chemical oxygen demand (COD) and total suspended materials (TSS) can be removed from sewage water using conventional methods and the proposed mechanism of removal is adsorption techniques by a copolymer of iron and aluminum impregnated with activated silica derived from rice husk [10,11].

The grey water reuse has great potential in wide range of applications in domestic, commercial, institutional as well as industrial buildings [12].

Coagulants destabilize colloids and dissolved constituents by a combination of six mechanisms; these based on compression of the double layer [13,14], counter ion adsorption and charge neutralization [15], enmeshment in a precipitate [16], heterocoagulation [17].

The aims of current research concern treatment of grey water in a two stages 1st conventional technique of water treatment (coagulation, flocculation, sedimentation, and filtration) using different of Poly inorganic coagulants such as, poly aluminum chloride silicate (PACSi), poly aluminum hydroxy sulphate silicate (PAHSSi), poly ferric chloride silicate (PFeClSi), and poly aluminum ferric chloride silicate (PAIFeClSi) through coagulation/flocculation/sedimentation and filtration steps as coagulant to reduce organic load followed by adsorption through anthracite to remove residual organic material.

2 Experimental Methods

2.1 1st step; (batch experiments):

All chemicals used in the study are lab trade. Anthracite (An) was kindly supplied by the Holding corrected for Water and Wastewater. Four of the highly effective modified natural materials such as poly aluminum chloride silicate, poly ferric chloride silicate, poly aluminum hydroxy chloride silicate, poly aluminum ferric chloride silicate, as well as modified natural material, and modified agro-residues were prepared and characterized as illustrated in the literatures [9 - 11]. They were applied for the reduction of BOD, COD and TSS from sewage waste water to obtain the water quality for re-use in agricultural drip irrigation systems, where, the treatment depends on precipitation, coagulation and adsorption techniques by poly inorganic coagulants (PIC). Each sample was mixed with 5-10 ppm of PIC and agitated rapidly for 1 (300 rpm), followed by slow mixing for five minutes and (30 rpm) and 20 minutes standing time.

The concentrations of pollutants were measured in raw samples and in the filtrate using AAS and analyses according to APHA 2005 [18]. The removal of the suspended materials will be achieved through the

implementation of conventional treatment and the optimum dose was determined using the jar test, which is composed of six stirred beakers, which can be utilized to simulate mixing and settling conditions in a clarifier [11].

2.2 2nd step; Affecting Factors of Residual COD Adsorption

The removal of the residual organic materials was achieved through the implementation of adsorption experiments and the study of affecting factors as shown in table:(1).

Table 1 : Affecting factors of residual COD adsorption.

| Factors | Conditions | | | | | | | |
|-----------------------|------------|-----|-----|-----|-----|-----|----|----|
| pH | 2 | 3 | 4 | 5 | 7 | 8 | 9 | - |
| Dose (g) | 2 | 4 | 8 | 12 | 14 | 16 | 18 | 20 |
| Contact time (min.) | 15 | 30 | 45 | 60 | 90 | 120 | | |
| Temperature (k) | 298 | 303 | 308 | 313 | 318 | - | | |
| Agitation forth (RPM) | 50 | 100 | 150 | 200 | 250 | 300 | | |

2.3 3rd step (column system)

The aim of the column test is to detect the parameters which are required to design industrial scale fixed bed adsorption columns, these parameters shown in table (3). Column study carried out by fabrication of column system with diameter 2 cm and height is 10 cm, the rate flow of COD solution is (5 ml/minute), grain size is 0.25 mm and the bed weight is 2g.

The experimental results were analyzed and breakthrough curves (Fig.4) were drawn for both bed weights of 2g (0.3 mm) 20 mg/L initial COD concentration. The corresponding bed height was 10 cm. the test was conducted under uniform flow rate of 5 mL/min. practically the concentration of the solution leaving the column changes very slowly when it is near to solution initial concentration. Thus it is assumed that the bed exhaustion is achieved at almost infinite time. Each constants determined using the equation which summarized in table (3)

3 Results and Discussion

3.1 The Main Conventional Stage

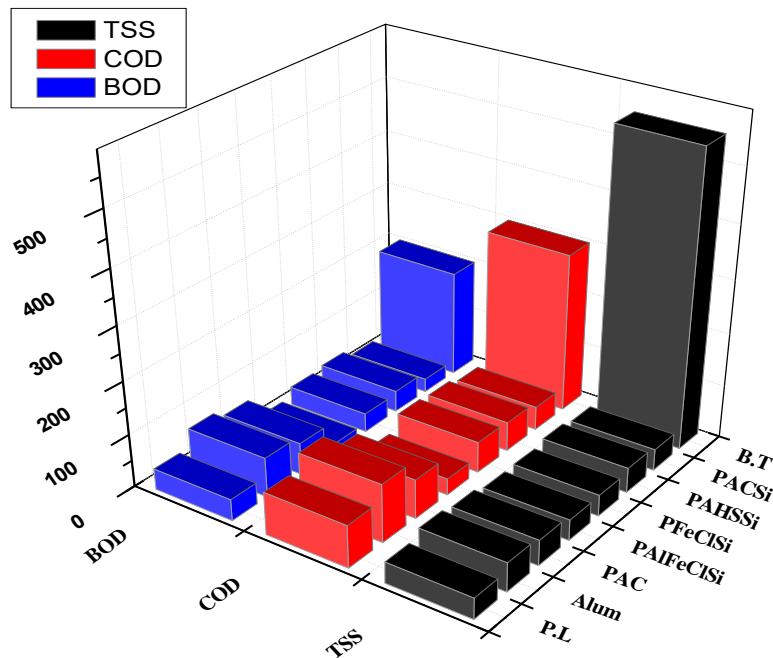
Table (2) and represented in Figure (1) summarize the variations of COD, BOD and TSS of grey water before and after treatment using four samples of poly inorganic coagulants based on silica and conventional Alum and PAC. It was found that, the removal percentages of TSS, COD and BOD are 90, 80.5, 72.3% respectively, using PACSi, and 90.2, 83.3, 71.1 %, respectively, using PAIHSSi, and 91.1, 83.2, 71.9 %, respectively, using PFeClSi, and 94.2, 89.2, 76.9 % respectively, using PAIFeClSi, and 88.2, 77.6, 43.4 %, respectively, using PAC, and 72.5, 54.4, 44.6 %, respectively, using Alum, and the maximum removal of TSS, COD and BOD are 94.2, 89.2, 76.9% respectively, using PAIFeClSi. PAIFeClSi is the superior poly inorganic coagulant copolymer that may be attributed to the co-polymers of Si⁴⁺, Al³⁺ and Fe³⁺ [11].

Saleh et al ., 2019 concluded that, Alum coagulation followed by batch and continuous AC adsorption tests recorded that the optimal overall removal of turbidity (95.8%), COD (31.6%) and BOD (50.0%) were achieved at 20 mg/L of alum dose[19], whereas Shivangi et al ., 2017 concluded that COD reduced by 35-45%, BOD was reduced by 50-60% at a coagulant concentration of 10 mg/l [20].

As for the total dissolved solids, it increased due to the formation of new of spiked ions from addition of dissolved aluminum and iron derivatives such as, poly aluminum chloride silicate (PACSi), poly aluminum hydroxy sulphate silicate (PAHSSi), poly ferric chloride silicate (PFeClSi), and poly aluminum ferric chloride silicate (PAIFeClSi). No considerable exchange of pH and total dissolved solids occurred and all the values after treatment are within permissible limits according to Egyptian law 44/2000 [21].

Table 2: Variations of some parameters of grey water samples by the influence of different inorganic coagulants.

| coagulant Parameter s | Before treatment | After treatment | | | | | | Permissible limits |
|-----------------------------|---------------------|-----------------|--------|---------|-----------|------|------|-----------------------|
| | | PACSi | PAHSSi | PFeClSi | PAIFeClSi | PAC | Alum | |
| TDS mg/l | 750 | 760 | 790 | 780 | 770 | 775 | 778 | < 2000 |
| TSS mg/l | 350 | 35 | 34 | 31 | 25 | 41 | 45 | < 40 |
| pH | 7.5 | 7.21 | 7.17 | 7.3 | 7.11 | 7.09 | 7.13 | 6.0-9.0 |
| COD mg/l | 210 | 41 | 35.1 | 31.5 | 22.5 | 47 | 96 | < 80 |
| BOD mg/l | 80 | 22.1 | 23.1 | 22.5 | 18.5 | 45.3 | 44.7 | < 40 |

**Fig.1:** Variations of some parameters of grey waste water samples by influence of different Inorganic coagulants.

3.2 Additional Treatment; Batch study

3. 2.1. Affecting Factors

3.2.1.1. Effect of pH

The adsorption outline of organic carbon as COD by anthracite at several selected pH (3- 9) is demonstrated in (fig.2). At the pH from 3 to 9, the organic carbon adsorption was very rapid (25 to 98%). Therefore, pH 4 was the optimum pH and appropriated for all the adsorption studies to verify equilibrium.

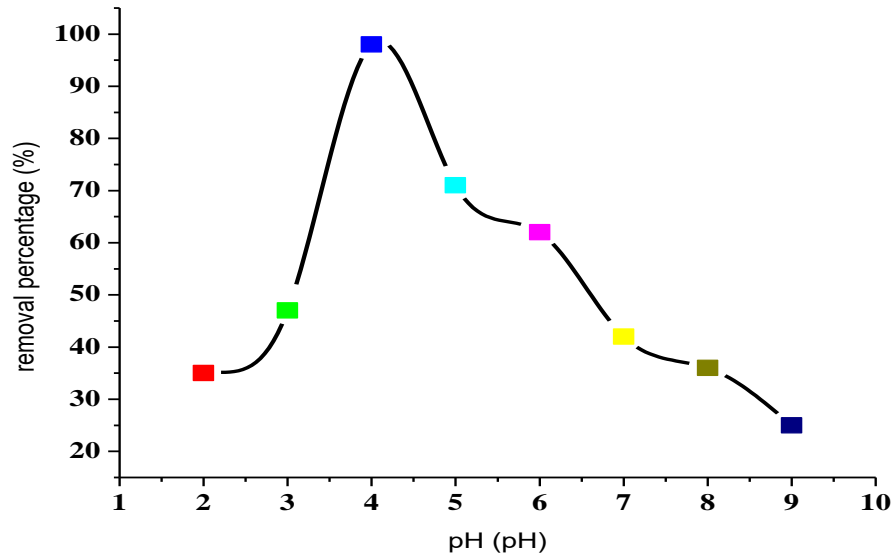


Fig.2: variation of removal percentages of COD versus Ph.

3.2.1.2 Effect of Agitation Time

The adsorption outline of organic carbon as COD by anthracite at several selected irritation time (15- 120 min) is demonstrated in (fig.3). At the irritation time duration from 5 to 90 min, the organic carbon adsorption was very rapid (20 to 99%). Therefore, this irritation time (90 minutes) was the optimum irritation time and appropriated for all the adsorption studies to verify equilibrium accomplishment

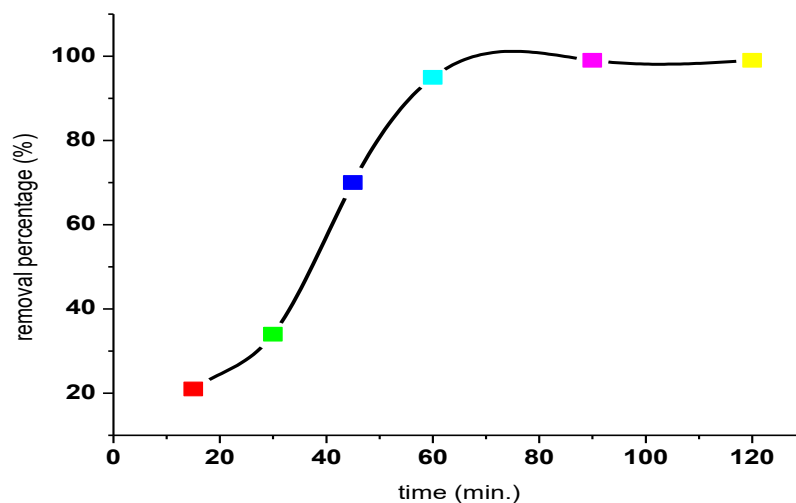


Fig. 3: variation of removal percentages of COD versus agitation time.

3.2.1.3. Effect of Adsorbent Mass

The adsorption outline of organic carbon by different selected masses (2 – 20 mg) of the An was demonstrated in fig.(4) Increasing the An mass from 2 to 20 mg, was accompanied with an increase in COD uptake from 2 to 99%. Therefore a dose 18 mg was the optimum dose and appropriated for all the adsorption studies to verify equilibrium accomplishment.

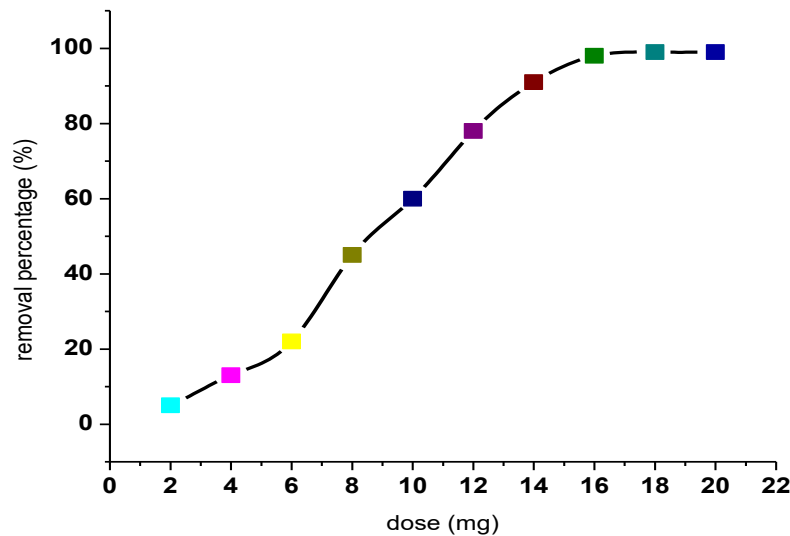


Fig. 4: variation of removal percentages of COD versus dose.

3.2.1.4 Effect of Agitation Speed

The adsorption profile of COD at different agitation speed (25 to 250 rpm) was implemented and demonstrated in (Fig.5), at the agitation speed period from 25 to 250 rpm, COD uptake removal percentage was rapidly increased from 16 to 99.0%. Therefore agitation speed (200 rpm) was the optimum agitation speed and appropriated for all the adsorption studies to verify equilibrium accomplishment.

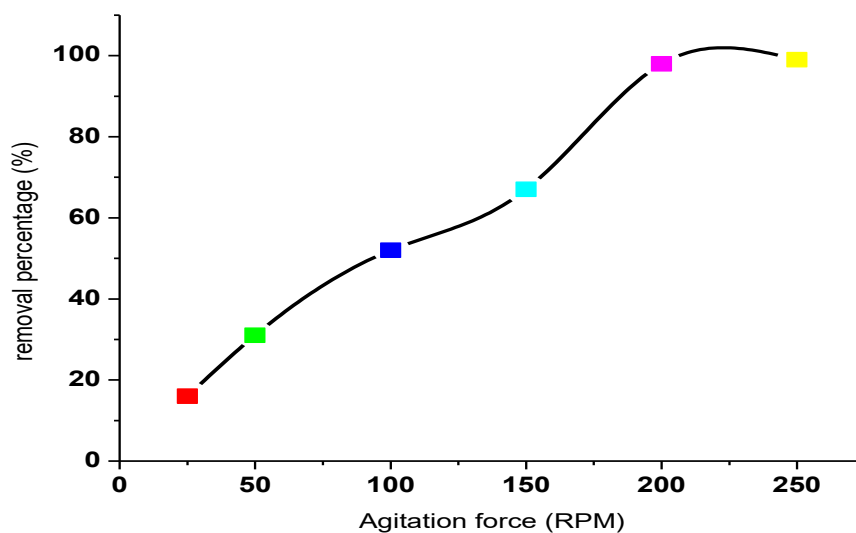


Fig.5: variation of removal percentages of COD versus RPM.

3.2.1.5. Effect of Temperature

Temperature has important effects on the adsorption process. The effect of temperature on the adsorption isotherm of the organic carbon as COD on anthracite was studied at 298, 305, 308, 313 and 318 K; the results were displayed in fig. (6). the results revealed that the removal percentage decreased from (98 to 76 %) with temperature increase from 298 to 318 K. This decrease in the removal percentage with temperature is due to the enhancement of the desorption step in the adsorption mechanism indicating that the process is exothermic

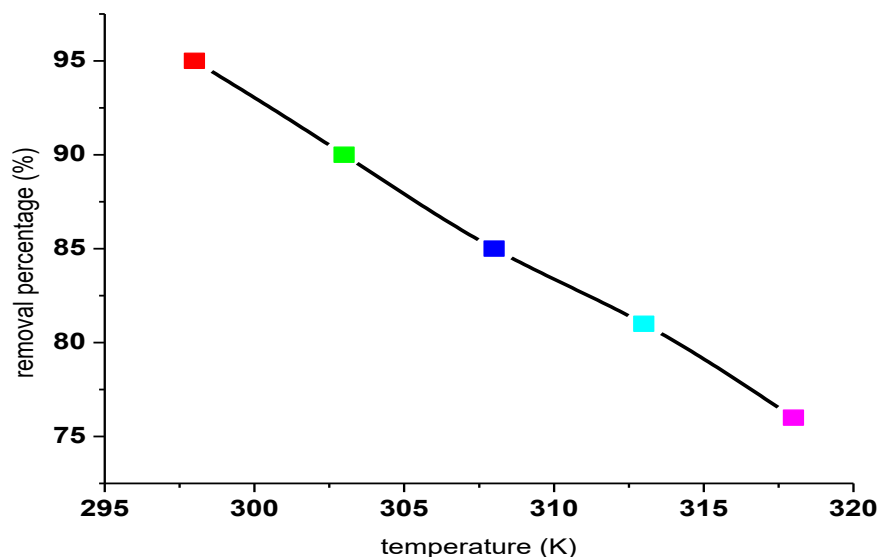


Fig.6: variation of removal percentages of COD versus temperature (K).

The optimum condition of all these factors can be summarized as follows: pH 4, agitation time is 90 minutes; temperature is 298 K; dose is 18 mg/l for 99% removal efficiency of COD. Whereas Saleh et al., 2019 concluded that, the overall removal efficiencies were enhanced by activated carbon adsorption for COD up to 70.8% and BOD up to 57.2% at 20 min adsorption equilibrium time with 0.2 g/L of optimal AC dose [22].

3.3. Column Study (clean up stage)

The column system fabricated by a burette of a diameter 1 cm and a height 5cm. The adsorption capacity (q) at $C_0/C^\circ = 50\%$ of removal or $Q_{0.5}$ was 15.625 mg COD per 2 gram anthracite. The total integrated amount of COD adsorbate was divided by the weight of the sorbent placed in the column to yield the adsorption capacity of 31.25 mg/gl (fig.7). As shown in Fig.(8), the column was recycled at least five times and only needed 5 ml of HCl (1M) for each regeneration and no loss in sorbent mass per cycle was considered as shown in (fig.9) without affecting the sorption capacity of An for COD (with an initial concentration $C^\circ = 20$ ppm).

The increase of adsorption percentages of acid regeneration is due de-protontion of An. Such results are suitable for the elimination of residual COD.

To calculate bed capacity at breakthrough time, it can be determined by equation [23]

$$BC = G(C_0 - C) T_b$$

Where G = flow l/min., C_0 initial concentration, c concentration at T_b , T_b breakthrough time, ideal break through curve have been drawn in fig ()

To calculate bed capacity at infinite time, it can be determined from the following equation

$$BC = G(C_0 - C^*) T_b$$

Where G = flow l/min., C_0 initial concentration, c^* concentration at T_s , T_s infinite time, LUB length of unused bed can be determined from the following equation

$$LUB = Z/T_s (T_s - T_b)$$

Where Z is thickness of bed (cm), T_s infinite time, and T_b breakthrough time all data summarized in the table (3)

Table 3: results adsorption of organic material as COD onto Anthracite.

| Adsorbent (weight in g) | T _s (min.) | T _b (min.) at C/C ₀ = 0.125 | LUB | Amount adsorbed at T _b | | Amount adsorbed at T _s | |
|-----------------------------|--------------------------|--|------|-----------------------------------|-------|-----------------------------------|-------|
| | | | | Total (mg) | mg/g | Total (mg) | mg/g |
| 2 | 285 | 83 | 7.08 | 8.3 | 3.631 | 28.5 | 14.25 |

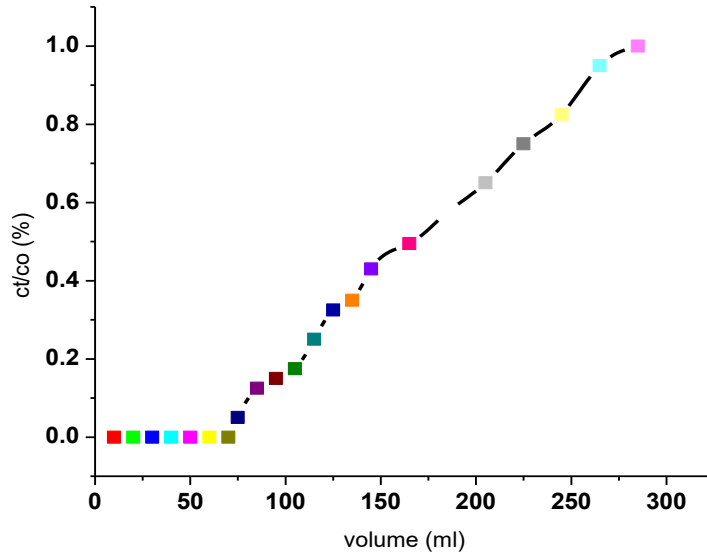


Fig. 7: C_t/C₀ versus integrated volume of organic carbon.

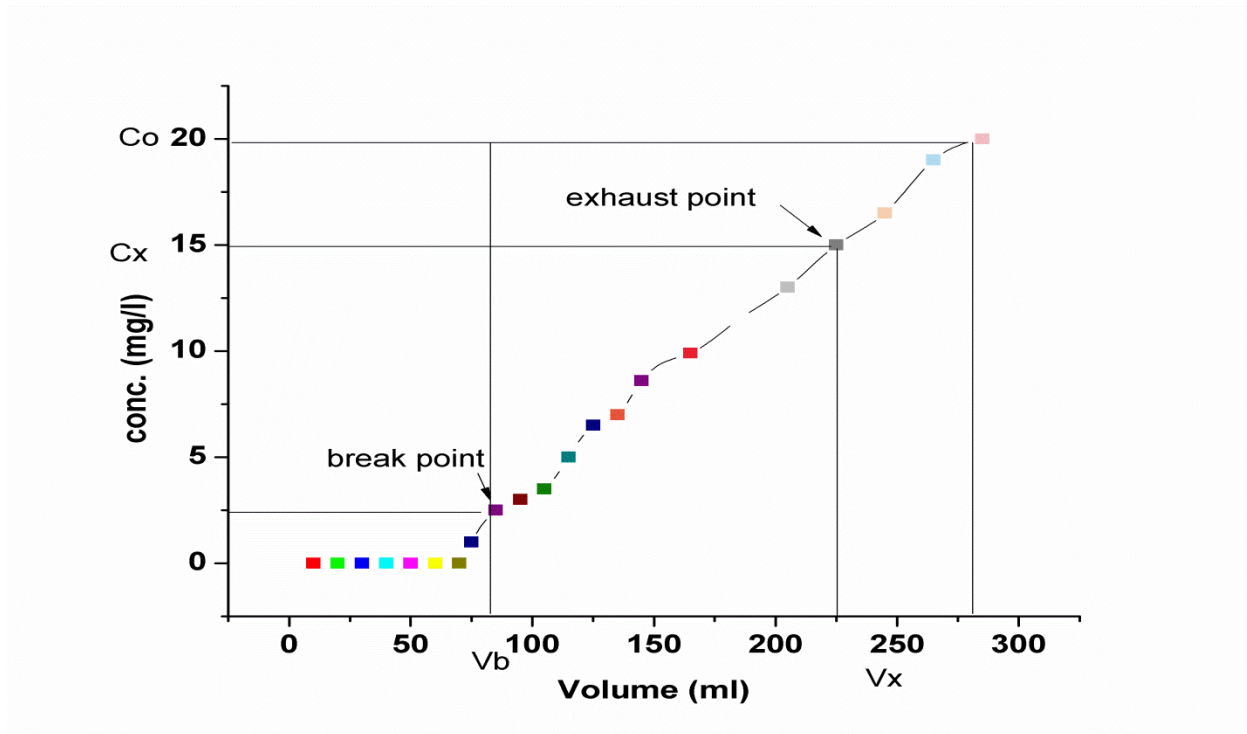


Fig.8: Adsorption wave of organic carbon onto An.

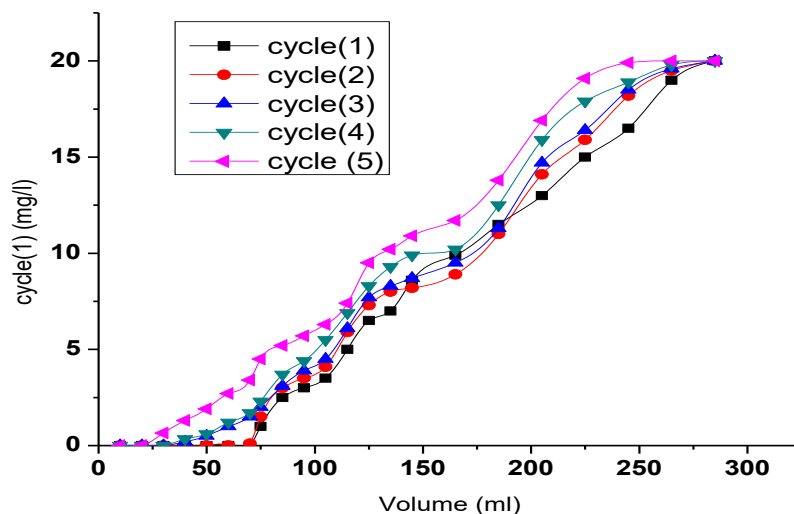


Fig.9: Desorption curve of an using H₂SO₄.

3.4 The Silica Rule for COD Reduction

The ion-exchange reaction on the silica surface is accomplished through the substitution of protons of the surface silanol groups by the metal ions from the solution.

Most particles in water, mineral and organic, have electrically charged surfaces, and the sign of the charge is usually negative. In the activated silica case, two important processes for producing this charge are considered in the following discussion:

First, surface groups on the solid may react with water and accept or donate protons. For an oxide surface such as silica, the surface site might be indicated by the symbol SiOH and the surface site ionization reactions take place.

Second, surface groups can react in water with solutes other than protons. Again, using the SiOH surface groups of silica.

These surface complex formation reactions involve specific chemical reactions between chemical groups on the solid surface (e.g., silanol groups) and adsorbate materials (e.g., organic materials). Surface charge is again related to solution chemistry, where Mⁿ⁺ = organic materials with n+ charge, (≡ SiOH) = silanol group on the SiO₂ surface and xH⁺ = number of protons released. The cation-exchange mechanism is expected with organic materials as the sorbent contains large amounts of silica as shown in table (4)..

Also, since the electrostatic attraction was possible negatively charged adsorbent surface and positively charged metal ion species, it seems that some electrostatic forces were involved in the adsorption process [23].

Table (4): Summarization of silica rules equations.

| Equation | Equation no |
|--|-------------|
| $\overline{\text{SO}}^- + \text{H}^+ = \overline{\text{SOH}}$ | (1) |
| $\overline{\text{SOH}} + \text{H}^+ = \overline{\text{SOH}}_2^+$ | (2) |
| (SOH represents a singly protonated oxide site) | (3) |
| cation adsorption (organic matters) reaction: | |
| $\overline{\text{SOH}} + \text{M}^{n+} + m\text{H}_2\text{O} = \overline{\text{SO.M(OH)}}_m^{n-m-1} + (m+1)\text{H}^+$ | (4) |
| anion adsorption (sewage water treatment) reaction: | |
| $\overline{\text{SOH}} + \text{A}^{n-} + m\text{H}^+ = \overline{\text{SOH}}_{m+1}\text{A}^{m-n}$ | |
| M and A represent cationic and anionic organic matters, respectively. | |

4 Conclusions

The maximum removals of TSS, COD and BOD are 94.2, 89.2 and 76.9 %, respectively; using Poly aluminum ferric chloride silicate (PAIFeClSi) which is suggestive as a favorable coagulants in different treatment technology compared to conventional coagulants. The superiority of poly in-organic coagulant copolymer may be attributed to the copolymers of Si^{4+} , Al^{3+} and Fe^{3+} . Therefore the developed rice husk co-agulants are considered as a better replacement technology for grey water treatment due to low cost and good efficiency in this application. The residual organic carbon was removed by the column system using anthracite as adsorbent materials. The optimum condition for the removing of 99% from the residual COD was in acidic medium (pH 4), agitation time 60 minutes, temperature is 298 K and dose is 18 mg/l.

The results of column system showed that the adsorption capacity (q) at ($C_t/C_o = 50\%$ or $Q_{0.5}$) was 16.625 mg the column recycled at least five times and only was 5 ml of HCl (1M) for each regeneration and no loss in sorbent mass per cycle should be considered. Increases of adsorption percentages of acid regeneration over acid regeneration due de-protonation of An. Such results are suitable for removal of residual COD.

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5 Nomenclature list

| Abbreviation | Item |
|--------------|---|
| AAS | Atomic absorption spectroscopy |
| Alum | Aluminum sulphate |
| An | Anthracite |
| APHA | <u>American Public Health Association</u> |
| B.T | Before treatment |
| BOD | Biological oxygen demand |
| C_o | Initial concentration of adsorbate |
| COD | Chemical oxygen demand |
| C_t | Concentration of adsorbate after time (t) |
| HCl | Hydrochloric acid |
| k | Kelvin |
| M | Molarity |
| min. | Minute |
| P.L | Permissible limit |
| PAC | Poly aluminum chloride |
| PACSi | Poly aluminum chloride silicate |
| PAHSSi | Poly aluminum hydroxy sulphate silicate |
| PAIFeClSi | Poly aluminum ferric chloride silicate |
| PFeClSi | Poly ferric chloride silicate |
| PIC | Poly inorganic coagulants |
| ppm | Part per million |
| Q_t | Adsorption capacity at time (t) |
| $Q_{t0.5}$ | adsorption capacity at $C_o/C_e = 0.5$ |
| RPM | Revolution per minute |
| TDS | Total dissolved solids |
| TSS | Total suspended solids |

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