Application of synthetic and grafted polymeric flocculants in agricultural wastewater treatment

Mohamed N. Ali $^{1*}$, Ahmed S. Fahmy$^{2}$, Rehab M. Elhefny$^{3}$

$^{1}$ Associate Professor of Environmental and Sanitary Engineering Department, Faculty of Engineering, Beni-Suef University, Egypt
$^{2}$ Civil Engineering Department, Faculty of Engineering in Shoubra, Benha University, Egypt
$^{3}$ Associate Professor of Environmental and Sanitary Engineering Department, Faculty of Engineering in Shoubra, Benha University, Egypt

*corresponding author: dr.mohamednabil8080@gmail.com

Abstract

Due to the large amounts of freshwater consumed in Egypt by the agricultural sector that is more than 85% of Egypt share of freshwater in addition to the high concentrations of salts, chemicals and nutrients produced from fertilizers. Reduction of these pollutants concentrations to an acceptable level and breaking the sedimentation stability of colloidal substances and organic particles for reuse for irrigation purposes was associated with the application of biological treatment with coagulants addition. The flocculation process was performed by using polydiallyldimethylammonium chloride (polyDADMAC) and polyacrylamide grafted oatmeal (OAT-g-PAM). The scale-pilot consists of an aeration tank equipped with an air blower, sedimentation tank followed by a filtration stage through 20 cm of pottery scrubs media. To study the performance of synthetic and grafted polymeric flocculants, 3 trials were performed. Activated sludge process without adding any polymeric flocculants was the control trial. In the second trial, polyDADMAC was added with a dose of 5 mg/l. Finally, OAT-g-PAM with a dose of 1.25 mg/l was used in the third trial. The physicochemical properties of agricultural wastewater were measured at the national research center in Cairo. It was found that OAT-g-PAM incorporated with activated sludge process was the most effective in treating agricultural wastewater as it achieved COD, BOD, TKN, TP, and TSS removal efficiency up to 92.29%, 93.13%, 90.64%, 90.46%, and 92.5%, respectively which made it suitable to reuse for agricultural purposes, in addition to its ability to biodegrade, environmentally friendly, and low dosage required compared to polyDADMAC.

Keywords: agricultural wastewater, synthetic polymeric, grafted polymeric, polyDADMAC, OAT-g-PAM.

Introduction

During the last 20 years, Egypt has been suffered from unequal water distribution, abuse of freshwater resources, and ineffective irrigation methods that were the main reason for the water scarcity in Egypt. Also, pollution and massive industrial development are factors causing a leak of water availability that leads to a decrease in Egypt's share by 7 billion cubic meters. In order to find alternatives for conserving freshwater resources and increasing the possibility of providing additional freshwater supply. Treatment of agricultural wastewater was the key solution of all the previous problems facing Egypt due to the large
amount of water consumed in the agriculture sector which reaches more than 85% of Egypt share of freshwater (Ministry of Water Resources and Irrigation, Arab Republic of Egypt, 2005).

The agricultural wastewater produces from the excess water that flows over the agricultural land through the submerged areas during the irrigation process; also it produces from plants processing crops irrigation. The agricultural wastewater has serious environmental effects if it was discharged directly into the surface water as this type of water contains large amounts of salts, chemicals and nutrients produced from fertilizers that used in order to maximize and improve crops production (Bratby, 2008). Besides the previous pollutants, agricultural wastewater is saturated with organic containments from food processing plants that need an expensive and high-consumed energy treatment to prevent the polluted water from running off through the ground and drained through the surface freshwater due to its high strength pollutants found in it (Cicek, 2003).

According to the guidelines of Law 48 year 1982 for the use of wastewater in agriculture and aquaculture. To control the critical hazards associated with wastewater reuse, Physical, chemical, and biological treatment must be applied to the agricultural wastewater in order to reduce the physicochemical characteristics of wastewater below the allowable limitations. Several biological treatment technologies could be used, such as activated sludge method (AS) (Sperling et al., 2014), sequence batch reactor (SBR), moving bed biofilm reactor (MBBR) (Hegazy et al., 2013), and integrated fixed film activated sludge (IFAS) (Zahari et al., 2019). Due to the accumulation of microbiological containments, heavy metals in biological from, and the chemical substances of crops and fertilizers in the agricultural wastewater, the most effective biological treatment method could be used is the activated sludge process (ASP) because it is characterized by several advantages compared to the other methods such as its low cost, high quality of effluent wastewater, and its hygienic operation due to few pests and odors involved (Sperling et al., 2014).

Despite the remarkable advantages of activated sludge process, ASP is not effective in removing the colloidal particles soluble in water due to its stability. In order to break the sedimentation stability of colloidal substances, dissolved salts, metals, and organic particles, several technologies could be used in order to make these particles with heavier mass and ready for settling (Brathy, 2006). Many methods have been used to get rid of colloidal substances, such as ion exchange, chemical precipitation, membrane filtration, and flotation. But coagulation and flocculation are seemed to be the most effective methods among these various technologies (Metcalfe et al., 2014).

Several types of coagulants and flocculants could be used to remove colloidal particles, such as inorganic coagulants, synthetic polymeric flocculants, natural polymeric flocculants, and grafted polymeric flocculants. Nowadays, depending on inorganic coagulants such as iron and aluminum salts has been decreased due to its low efficiency and production of metal by products that lead to environmental problems due to the produced toxic sludge (Lee et al., 2014). Natural polymers are moderate efficient with low short age because their compounds will biodegrade with time that produces very small flocs (Singh et al., 2006). Using synthetic polymeric flocculants has several advantages, such as its high flocculation efficiency with low dosage but its biodegradability and unfriendly effects on aquatic life make it undesirable (Lee et al., 2014). So searching for a polymer to combine the advantage of natural and synthetic polymers became an essential aim. So, using grafted polymers as flocculants is the win-solution to improve the removal efficiency of colloidal particles without unfriendly effects to the environment by producing denser, stronger flocs that improve the sedimentation and filtration process (Lee et al., 2012). Caustic grafted polymers are able to neutralize the negative charge of the suspended particles and bridge them. The bridging mechanism occurred when the polymer absorbed by the surface of the colloidal particles to form long loops and tails extended in all direction leading to the strong interaction between one particle to another and form strong and dense flocs without the need for pH adjustment leading to less produced sludge due to the strong bridging process. Grafted polymers have the ability to form a strong network between colloidal substances that leads to more effective flocs formation without the need for excessive dosages compared to the natural and synthetic polymer.

Several grafted polymers are synthesized by several ways, such as polymethylmethacrylate grafted psyllium, polycrylamide grafted starch, and Polyacrylamide grafted oatmeal. Selection of the suitable type of polymer depends on the characteristics of wastewater and the charge of the surface of the colloidal particles suspended in wastewater in order to use caustic or anionic polymer to neutralize the suspended particles to form flocs (Lee et al., 2014; Bolto and Xie, 2019). Each one of the grafted polymers is suitable to specific type of water according to the water characteristics which control the selection of the suitable polymer for the treatment process. So the objectives of this study were to use polydiallyldimethylammonium chloride (polyDADMAC) as a synthetic organic polymer (Bolto et al., 1998), and Polyacrylamide grafted oatmeal (OAT-g-PAM) (Bharti et al., 2013) as a grafted polymer for the treatment of agricultural wastewater in addition to a biological treatment by using activated sludge process in order to study the performance of each polymer in removing the suspended solids and organic containments from the agricultural wastewater and reuse it for irrigation purposes.
Materials and Methods

Collecting of agricultural wastewater samples

Agricultural wastewater was collected from an agricultural land in Egypt. The collected agricultural wastewater was the excess water from irrigation process through flooded areas. Plastic containers were pre-washed with dilute water to avoid any negative effect on the agricultural wastewater characteristics. 25 containers with 20 liters in volume for each were used to collect wastewater samples then transferred to the national research center for the experiments.

Preparation of the dose of polydiallyldimethylammonium chloride (polyDADMAC)

The dose of polydiallyldimethylammonium chloride (polyDADMAC) was determined by using the gar test. That test was performed at the national research center in Cairo, Egypt. Six jars filled with one liter of agricultural wastewater for each were used to determine the required dose of polydiallyldimethylammonium chloride (polyDADMAC). Different doses (1; 2; 3 and 4mg/L) of polyDADMAC were added to each jar in order to choose the suitable dose. In order to achieve a complete mix between agricultural wastewater and polyDADMAC, a mixer was operated at 100 to 150 rpm for a minute and then followed by a flocculation process at 30 rpm for 15 to 20 minutes for the slow flocs growth stage. The mixers were turned off to allow flocs to settle for 50 minutes, then the final residual turbidity in each jar at the end of settling duration was measured to determine the suitable dose. Figure 1 shows the molecular structure of polydiallyldimethylammonium chloride (polyDADMAC).

Materials required for synthesis of polyacrylamide grafted oatmeal (OAT-g-PAM)

In order to prepare the ingredients necessary for the Synthesis of grafted copolymer. Acrylamide, Ceric ammonium nitrate, and acetone were required. All chemicals were obtained from the (National for Chemical Industries-NASYDCO) in Egypt.

Synthesis of polyacrylamide grafted oatmeal (OAT-g-PAM)

Depending on the ceric ion induced redox method, reactions required for grafting process were performed as followings:

- A container of 40 ml of distilled water was prepared, and then 1 g of oatmeal was added to the water.
- By continuous and constant stirring, oatmeal was dissolved into the distilled water to form a solution.
- 10 grams of acrylamide was added to the previous solution with proper batching to ensure the complete mixing with the solution.
- Borosil beaker with 250 ml in volume was prepared to contain the reaction.
- The prepared solution of 10 grams of acrylamide was transferred into the Borosil beaker.
- The oxygen-free nitrogen gas was purged through the solution mixture.
- 0.1 grams of catalytic amount of ceric ammonium nitrate was added to the mixture and the process of nitrogen purging was still continuous.
- At room temperature (25), the reaction was continued for one day.
- The precipitate of graft copolymer was subjected to a high temperature in a hot oven in order to dry it.
- The resulting grafted polymer was ground and sieved to get ready to use.
- Acetone was used in order to extract any occluded polyacrylamide produced from the previous reaction.
- The final molecular structure of polyacrylamide grafted oatmeal (OAT-g-PAM) is shown in Figure 2.

Description of the experimental pilot

In this study, an activated sludge scale-pilot was established as a simulation of the biological treatment process, as shown in Figure 3. The scale-pilot consists of three rectangular glass tanks of 54 liters in volume in addition to a large perforated bottle with filter media.
of 20 cm thickness. In order to avoid using a pump due to its produced pressure that affects negatively to the treatment process, the pilot tanks were located at 3 different levels to facilitate transportation of water from one tank to another under gravity. The description of the pilot was mentioned in Table 1. The raw wastewater was fed into the aeration tank in order to remove any organic compounds from agricultural wastewater by the action of microorganisms. The aeration tank was equipped with a double-outlet air blower (portable cf0023) to supply the sufficient oxygen required for the bio-degradation process. The Agricultural wastewater remained in the aeration basin for hydraulic retention time of 8 hours to give the bacteria sufficient time to grow and get rid of all nutrients and organic containments soluble in wastewater. After a hydraulic retention time of 8 hours, wastewater was passed to the sedimentation tank to settle the by-products produced from the aeration tank. In order to increase the concentration of bacteria in the aeration basin, 25% of the settled sludge was returned to the aeration basin. The effluent of the aeration tank will be retained to settle in the sedimentation tank for 3 hours then passed through a perforated bottle with pottery scrubs media of 20 cm thickness to a collection tank where the treated water was collected. All samples were analyzed at the national research center in Cairo in order to study physicochemical properties of the agricultural wastewater. In this study, 3 trials were performed as the following:

- 8 hours activated sludge cycle followed with a filtration stage through a pottery scrubs media.
- 8 hours activated sludge cycle with 5 mg/l of polyDADMAC as a flocculant followed with a filtration stage through a pottery scrubs media.
- 8 hours activated sludge cycle with 1.25 mg/l of Polyacrylamide grafted oatmeal (OAT-g-PAM) as a flocculant followed with a filtration stage through a pottery scrubs media.

Table 1. The dimensions and the operational conditions of the pilot-scale.

| Treatment unit       | Parameters                      | Values        |
|----------------------|---------------------------------|---------------|
| Sedimentation tank   | Length × width × depth          | 60 × 30 × 30 cm |
|                      | Volume of water                 | 45 liters     |
|                      | Hydraulic retention time        | 3 h           |
| Aeration tank        | Length × width × depth          | 60 × 30 × 30 cm |
|                      | Volume of water                 | 45 liters     |
|                      | Hydraulic retention time        | 8 h           |
|                      | Sludge recirculation            | 25%           |
| Filtration bottle    | Thickness of filter media        | 20 cm         |
|                      | Type of filter media            | pottery scrubs media |
| Collection tank      | Length × width × depth          | 60 × 30 × 30 cm |
| Air blower           | Rate of flow                    | 320 l/h       |

Figure 3. Scheme of activated sludge pilot with polymers as a flocculant.
Chemical and physical analysis

- BOD, COD, TKN, and TP concentrations were performed at the national research center in Cairo according to standard methods for the examination of water and wastewater.
- Temperature was measured daily before taking wastewater samples from treatment stages.
- Determination of the polyDADMAC dose was measured by using a jar test at the national research center in Cairo.

Results and Discussion

Characteristics of raw agricultural wastewater

The raw agricultural wastewater samples were obtained from agricultural land in Egypt. In order to evaluate the concentrations of nutrients and organic matters compounds, samples were tested at the national research center in Cairo. The physicochemical properties of agricultural wastewater such as BOD, COD, TSS, TKN, and TP were illustrated in Table 2. The agricultural wastewater is this kind of wastewater produced from water runoff over fields, excess irrigation water and drainage water from farmlands. In addition to many farm activities such as animal feeding and operation of agricultural products. As shown in Table 2, the characteristics of the agricultural wastewater indicate high concentrations of pollutants. The pH of the raw agricultural wastewater was tended to neutralization as it ranges from 7.6 to 7.7 which agree with the pH values of other studies that range from 7 to 7.96 (Lu et al., 2010). As known, the pH of agricultural wastewater was affected by the soluble substances in soil, the type of crops and the fertilizers used in the farmland. All the previous factors have a neutral pH ranges from 6.5 to 7.5 that explained the neutralization of the effluent agricultural wastewater (Hatten and Liles, 2019). The concentration of the organic matters represented in COD and BOD values were 390 ± 56.6 mg/l and 175 ± 21.2 mg/l, respectively. The concentrations of the agricultural wastewater were seemed to be at the range of the average overall organic matter concentration of the agricultural wastewater that ranged from 89 to 433 mg/l (Rozema et al., 2016). The main source for these organic containments found in the agricultural wastewater was the fertilizers used to maximize the production of the crops in addition to the plant activity.

| Parameters                | Units | Raw water (1) | Raw water (2) | Average ± standard deviation |
|---------------------------|-------|---------------|---------------|------------------------------|
| pH                        | ---   | 7.7           | 7.6           | 7.65 ± 0.1                   |
| Biological Oxyge... demand, BOD | mg/l | 190           | 160           | 175 ± 21.2                   |
| chemical Oxyge... demand, COD | mg/l | 430           | 350           | 390 ± 56.6                   |
| Total Suspended Solids, TSS | mg/l | 140           | 110           | 125 ± 71.2                   |
| Total Nitrogen, TN        | mg/l  | 80            | 65            | 72.5 ± 10.6                  |
| Total phosphorous, TP     | mg/l  | 37            | 32            | 34.5 ± 3.5                   |

From Table 2, the concentrations of the TKN and TP were 72.5 ± 10.6 and 34.5 ± 3.5 mg/l (Table 2). In other studies, the TKN concentration of the agricultural wastewater was from 67 to 402 mg/l and from 2.6 to 235 mg/l for TP concentration (Vymazal and Kröpfelová, 2009; Rozema et al., 2016). By comparing the TP and TKN concentrations of the raw agricultural wastewater with the values from other studies. It seemed to be at the average overall range of TP and TKN concentration. Although the TP and TKN concentrations coincide with the others. It seemed to be small concentrations that were due to the high absorption ability of the plants roots. Due to the capability of plants roots to uptake nitrogen compounds from soil in addition to the participation of the soil in ammonium ion absorption, all these factors were the main reason for decreasing TN and TP concentration of the agricultural wastewater (Lee et al., 2009).

Results of treatment trails

In order to produce treated wastewater that could be used for irrigation purposes. Several treatment attempts were performed using polyDADMAC and Polyacrylamide grafted oatmeal (OAT-g-PAM) as flocculants. All the final effluents were compared with the Egyptian standards for reusing the treated wastewater in irrigation to ensure that it met the allowable limitations. The physicochemical characteristics of agricultural wastewater after each stage of treatment were shown in Table 3. From Table 3, it was clear that the 3-trials were effective for agricultural wastewater treatment in order to reuse for unrestricted irrigation purposes. All the effluent concentrations were below the TSS, COD, BOD, TP and TKN limitations according to Egyptian guidelines for unrestricted irrigation (ECP 501, Egyptian Code of Practice for the Reuse of Treated Wastewater for Agricultural Purposes, 2005).
But depending on Polyacrylamide grafted oatmeal (OAT-g-PAM) as a flocculant besides the activated sludge process was the most effective trial among all.

**Performance of polyDADMAC for total suspended solids removal**

In this study, polyDADMAC was added to the aeration tank with a dose of 5 mg/l in order to be used as a flocculation aids to reduce the concentrations of organic compounds, suspended and soluble solids found in agricultural wastewater. By depending on the direct flocculation process without the addition of any chemical coagulants, polyDADMAC was able to reduce the TSS with removal efficiency of 80.55%. That was due to the high molecular weight of polyDADMAC that contribute the flocs together through the bridging mechanism. As soon as the polyDADMAC was added to the wastewater, it caused an increase of the positive charge leading to neutralize the colloidal particles to form flocs (Ariffin et al., 2012). So the reduction of total suspended solids is related to the formation of flocs in addition to their amount and size.

| Parameters          | Raw | After Aeration | After Sedimentation | After Filtration | Removal efficiency % |
|---------------------|-----|----------------|---------------------|------------------|----------------------|
|                     |     | HRT= 8 h       | HRT= 3 h            |                  |                      |
| PH                  | 7.7 | 7.7            | 7.5                 | 7.5              | ----                 |
| COD                 | 430 | 165            | 92                  | 63               | 82%                  |
| BOD                 | 190 | 73             | 44                  | 32               | 80%                  |
| TSS                 | 140 | 113            | 50.6                | 27               | 75.45%               |
| TKN                 | 80  | 48.3           | 31.6                | 21               | 67.69%               |
| TP                  | 37  | 26.1           | 13                  | 6.7              | 79.06%               |
| PH                  | 7.6 | 7.4            | 7.5                 | 7.7              | ----                 |
| COD                 | 350 | 215            | 113                 | 64               | 81.71%               |
| BOD                 | 160 | 92             | 33                  | 31               | 80.63%               |
| TSS                 | 110 | 86             | 34                  | 21.4             | 80.55%               |
| TKN                 | 65  | 46             | 21.3                | 8.6              | 86.77%               |
| TP                  | 32  | 26.4           | 12.7                | 6.1              | 80.94%               |
| PH                  | 7.6 | 7.4            | 7.5                 | 7.5              | ----                 |
| COD                 | 350 | 172            | 98                  | 27               | 92.29%               |
| BOD                 | 160 | 76             | 24                  | 11               | 93.13%               |
| TSS                 | 110 | 69             | 29                  | 10.3             | 90.64%               |
| TKN                 | 65  | 42             | 20                  | 6.2              | 90.46%               |
| TP                  | 32  | 22             | 8.6                 | 2.4              | 92.50%               |

Several studies reported that using polyDADMAC as a direct flocculant was effective to reduce TSS and colloidal organic matter up to 90% (Zahrim et al., 2017). So the performance of polyDADMAC in this study was slightly lower than the results obtained by the others. As known, agricultural wastewater contains many soluble metals such as ferric ions (Fe$^{3+}$) obtained from the farm lands and crops. The presence of metal ions affects the bridging mechanisms of the polymers as it was reported in several studies, the ferric metals ion affected the performance of the flocculation process by polyDADMAC due to the reaction between metal ions and carboxylate groups on the polymer chain leading to a remarkable reduction in charge density of the polyDADMAC that affect the flocs formation through the bridging mechanism (Bolto and Gregory, 2007). Also, the destabilization of the flocs that occurred due to the excess cationic ions from polyDADMAC leading to the deteriorations of the flocs from the flocculation by polyDADMAC, the availability of excess cationic ions was a reason of the charge reversal that affected the flocculation process (Bolto and Gregory, 2007; Lee et al., 2014).

**Performance of polyDADMAC for total suspended solids removal**

Using polyDADMAC as a direct flocculant was effective in COD and BOD removal with removal efficiency up to 81.71% and 80.63%, respectively that was lower than the results reported by the other researchers. Several studies achieved organic matter removal up to 90% to 95% (Bolto et al., 1998). This reduction in organic matter removal efficiency may be due to the insufficient dosage polyDADMAC that caused decreasing of the positive charges that affected the neutralization mechanism leading to deterioration.
of the bridging process. Also, the toxicity of the synthetic polyDADMAC was a reason for the low organic matter removal efficiency. PolyDADMAC is a moderate toxic polymer that affected the performance of micro-organisms causing a decrease in the biodegradation of organic matter (Bolto and Gregory, 2007).

**Performance of polyDADMAC for nitrogen and phosphorous removal**

The TKN and TP removal efficiency in the case of using polyDADMAC as a flocculant was 86.77% and 80.94 %, respectively. Depending on organic polymer to reduce the TN and TP concentrations by primary precipitation process without performing biological treatment wasn’t sufficient. Many studies showed that the TN and TP removal efficiency was 8% and 12 %, respectively and increased up to 65% to 80% in case of following by biological treatment stage (Czerwionka et al., 2020). From the previous results, we concluded that using organic polymer such as polyDADMAC was not effective in nutrients removal without a biological treatment process. That was due to the formation of loose and weak flocs with low sedimentation properties. Also, the precipitation of colloidal and suspended substance has a negative effect on the nutrients removal and caused a disturbance in the nitrogen and phosphorus removal from the agricultural wastewater leading to a reduction rate of 20% to 40% for the TN removal and 40% to 70% for the phosphorus removal (Drewnowski and Makinia, 2014; Czerwionka et al., 2020).

**Performance of (OAT-g-PAM) for total suspended solids (TSS) removal**

Polyacrylamide grafted oatmeal (OAT-g-PAM) is a grafted polymer that combines the properties of both natural and synthesis polymers. From Table 3, polyacrylamide grafted oatmeal was able to remove the TSS by a removal efficiency of 90.64% (Table 3). This high removal efficiency was due to the higher flocculation efficiency of the grafted polymer. The percentage grafting of OAT-g-PAM was 917 % that caused an increase of the surface area of the grafted polymer leading to a high ability to link colloidal particles through flocs arrangement (Bharti et al., 2016) in addition to higher hydrodynamic volume and intrinsic viscosity reached to 1.77 dl/g which were a participate factors in flocculation capacity improvement. Several studies refer to the high efficiency of Polyacrylamide grafted oatmeal that achieved a reduction in TSS concentrations up to 90 % (Lee et al., 2014). From previous results, OAT-g-PAM was an effective flocculant than polyDADMAC as it achieved TSS removal efficiency up to 90.64% compared with 80.55% achieved by polyDADMAC. That was due to the longer polymer chain and the higher radius of gyration which allowed to the grafted polymer molecules to approve more configurations to form complex interactions with more than colloidal substances (Lee et al., 2014; Bharti and Mishra, 2016) and to increase the cooperative effects between bridging flocculation and charge neutralization.

**Performance of (OAT-g-PAM) for organic matter and nutrients removal**

More than 90% of the grafted polymers were able to remove COD concentrations up to 98% that was higher than the results achieved by polyDADMAC (Sinha et al., 2013). From Table 3, OAT-g-PAM was effective in removing about 92.29% COD, 93.13% BOD, 90.46% TKN, and 92.50% TP. By comparing these results with the effluents concentrations achieved by the other researchers. Many studies reported that the grafted copolymers used as direct flocculents were able to achieve COD removal up to 98 %, TN removal up to 53%, and TP removal up to 57 % (Ngema et al., 2020) without using a biological treatment process. This impressive removal efficiency of OAT-g-PAM was due to high molecular weight and intrinsic viscosity due to grafting chains process that allows for easy approachability of organic particles and nutrients in water ( Sinha et al., 2013; Ngema et al., 2020).

**Conclusion**

Agriculture sector consumes more than 85 % of Egypt share of freshwater that produces massive amounts of wastewater that could be reused after applying the proper type of treatment for unrestricted irrigation purpose. Application of activated sludge process with adding highly-effective flocculants such as organic polymer such as polyDADMAC and grafted copolymer such as Polyacrylamide grafted oatmeal was very effective in removing organic matter, suspended solids, and nutrients. Despite the high removal efficiency achieved by both polymer, Polyacrylamide grafted oatmeal seemed to be more effective than polyDADMAC due to its higher molecular weight, longer polymer chain, and higher intrinsic viscosity leading to pollutants uptake up to 90% in addition to its remarkable advantage such as its friendly effects to the environment that is not provided in polyDADMAC that produce toxic residues which affect the aquatic life.

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