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The Evaluation of the Performance of Polymeric Coagulants and Flocculants on The Characteristics of Paper Mill Effluent

Alireza Seify [1], Majid Peyravi[*] [2] Mehri Esfahanian [3], Hassan Ahmadi [1],

1: Department of Civil Engineering, Roudehen Branch, Islamic Azad University, Roudehen, Iran.

2: Associate Professor, Chemical Engineering Department, Babol Noshirvani University of Technology, Shariati Ave., Babol, Iran.

3: Department of Chemical Engineering, Qaemshahr Branch, Islamic Azad University, Qaemshahr, Iran.

Abstract: - Background and Aim: Due to the high pollutant load of paper mill effluent and to decrease contaminants, effluent should be treated before being discharged into the environment.

Materials and Methods: After collecting the samples of paper mill effluent in Babol city, the high levels of COD [Chemical Oxygen Demand] and TSS [Total Suspended Solids] were found. Therefore, polymeric coagulants, such as iron sulfate, ferric chloride, polyaluminum chloride, and alum were employed, as well as anionic and cationic polyacrylamide flocculants.

Results: Ferrous sulfate, ferric chloride, aluminum polychloride, and alum at their optimum pH removed 12%, 13.5%, 15%, and 23% of the effluent COD, respectively; as well as 45.5%, 47%, 49%, and 52% of TSS. Then, by examining the coagulants' concentration effect, alum with an optimal concentration of 1 g/l removed 23.7% COD and 56.4% TSS. Moreover, the effects of anionic and cationic polymeric coagulants were studied. Considering the results, using an anionic coagulant at a concentration of 0.004 g/l in combination with the optimal amount of alum resulted in eliminating 48.6% COD and 69.6% TSS.

Conclusion: Based on the results, coagulation and sedimentation methods can be utilized to treat the effluent of paper mills. Furthermore, using an alum coagulant and an anionic coagulant simultaneously improves the efficiency of the coagulation and flocculation processes to remove the pollutants from paper mill effluent.

***Keywords**: Chemical treatment, Coagulation and flocculation, Pulp and paper industry, Polymeric materials, COD, TSS.*

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1. Introduction

The rapid population growth, the increasing demand for facilities to meet human needs, and problems such as over-exploitation of existing resources have caused soil, air, and water pollution. Paper is an essential product in modern daily life. The global production of paper in 2015 exceeded 390 million tons, and it is expected that this trend will continue to increase annually [1]. Various methods are used for paper production, with the main types being mechanical, chemical, and thermal methods [2]. The amount of water consumption in paper mills varies depending on the type of paper produced and the process used. On average, water consumption in these industries ranges from about 2000 to 60000 gallons per ton of product, leading to the production of effluents with high pollution loads in various parts of the process. As a result, paper production factories account for 43% of the 3 billion tons of industrial effluent [1]. The main pollutants in the effluent from paper mills include high levels of chemical oxygen demand [COD], which is usually more than 10000 mg/l, and a large amount of total suspended solids [TSS] present in it [3]. The effluent produced is treated and recycled to conserve energy and reuse raw materials. Among the various technologies used for industrial wastewater treatment are adsorption [4], electrochemical [5], electro-Fenton [6], chemical coagulation [6], membrane processes [7], and sedimentation [8].

Pollutants from pulp and paper mills mainly include gases produced from the recycling process, the kraft pulping stage, sulfur oxides [SO2 and SO3] from lime kilns and recovery furnaces, and solid waste such as sludge produced in primary and secondary treatment sections, tree bark particles, and other wastes from the factory. The color of the effluent is due to the presence of organic compounds including hemicellulose, benzoic acid, phenol, benzophenone, lignin, and their degradation products, which are produced by the action of chlorine on lignin during the bleaching process, making them resistant to degradation and removal [9]. Lignin is a complex porous biopolymer made of phenylpropane units with carbon-carbon and ether bonds [10].

The dark brown color of the produced effluent is formed during the degradation of hemicellulosic materials, used as an indirect measure of the lignin compounds present in the outflow. The higher the amount of these compounds in the effluent, the darker its color and the greater its tendency to form foam. Studies have shown that the presence of these by-products in the effluent of paper production factories gives them toxic properties [11]. If this effluent enters the environment without any specific treatment system, even at very low concentrations, it can have adverse effects on living organisms [12]. The produced effluents are typically treated in industrial treatment plants, which generally include sections such as primary settling tanks, primary clarifiers, biological treatment systems, and secondary clarifiers, to remove suspended solids and dissolved organic materials.

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Additionally, for the treatment of effluent from wood and paper factories, several physical and chemical processes are used, including a combination of ultrafiltration and reverse osmosis processes, ion exchange, chromatography, lime precipitation, and adsorption with activated carbon [13-16]. Biological treatment is widely used to reduce the pollution load of industrial effluents and to degrade lignin in paper effluents [17]. Although biological treatment is not very efficient in removing the color of paper mill effluent [18], it results in a significant reduction in biochemical oxygen demand [BOD] and COD in this effluent. However, the use of this process always comes with problems such as high energy consumption, high sludge production [19], and operational issues such as foam production and clogging in secondary clarifiers [20]. Membrane-based technologies, including ultrafiltration [21] and electrochemical membrane reactors [5], have also been used with limited success for the separation of pollutants in paper mill effluent. The coagulation and flocculation process, using materials such as alum, ferric chloride, polyaluminum chloride [PAC], and lime, can be considered an effective treatment method for reducing COD, TSS, and color in effluents [22].

Lee et al. used pretreatment methods including coagulation and Fenton before ultrafiltration [UF] to investigate their effects on reducing UF fouling for treating secondary effluent from a recycling paper mill. Results showed that the presence of pretreatment methods significantly improved UF performance. After Fenton pretreatment, there was less membrane fouling and greater removal of organic matter. It is noteworthy that the UF effluent after Fenton pretreatment can fully meet the water reclamation requirements for industrial uses. In comparison, Fenton and UF had better performance in reducing dissolved organic matter [23]. Grötzner et al. investigated the efficiency of a combination of physical and chemical pretreatment with the coagulation-flocculation-sedimentation [CFS] process and advanced oxidation process [AOP] by Fenton for treating pulp and paper mill effluent. Results showed that CFS had better efficiency in removing solid matter, and Fenton was better at removing recalcitrant compounds like lignin. The combined CFS and Fenton process achieved a 61% removal efficiency for COD and 76% for lignin content [8].

Ansari et al. studied the performance of a full-scale coagulation-flocculation/dissolved air flotation [DAF] system as a pretreatment technology for enhancing the biodegradability of high-strength waste paper recycling wastewater. Optimal doses of coagulant and flocculant were determined using jar tests. The optimal doses were 1500 mg/l of polyaluminum chloride as a coagulant and 40 mg/l of cationic polyacrylamide as a flocculant [24].

The effluent from paper mills generally contains a complex mixture of various organic compounds such as carbohydrate degradation products, lignin, and extractives. These organic compounds vary chemically, and it is not surprising that the nature of these compounds significantly affects the choice of coagulant used. Moreover, the nature of the effluent produced in these mills can vary depending on the type of product produced, the

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process used, and the equipment employed. Therefore, each paper mill requires an assessment of the treatability of its effluent to determine the appropriate treatment processes. In this study, the paper mill effluent from the Babolkanar industrial estate in Babol city was examined for its treatability using coagulation and flocculation processes to determine the best coagulant and flocculant, and to establish the operational conditions such as the appropriate pH and the amount of each substance to achieve the highest removal rates of COD and TSS, using these materials as a chemical pretreatment system.

This significant reduction in effluent. However, the use of this process always comes with problems such as high energy consumption, high sludge production [19], and operational issues like foam production and clogging in secondary clarifiers [20]. Membrane-based technologies, including ultrafiltration [21] and electrochemical membrane reactors [5], have also been used with limited success for the separation of pollutants in paper mill effluent. The coagulation and flocculation process is one of the best options for treating paper mill effluent using coagulants like alum, ferric chloride, lime, iron sulfate, and polyaluminum chloride [PAC] [22]. It can be considered an efficient treatment method for reducing COD, TSS, and effluent color.

Lee et al. used pretreatment methods, including coagulation and Fenton, before the ultrafiltration [UF] process to investigate their effects on reducing UF fouling for treating secondary effluent from a recycling paper mill. Results showed that the presence of pretreatment methods significantly improved UF performance. After Fenton pretreatment, there was less membrane fouling and greater removal of organic matter. It is noteworthy that the UF effluent after Fenton pretreatment can fully meet the water reclamation requirements for industrial uses. In comparison, Fenton and UF had better performance in reducing dissolved organic matter [23].

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2. Operational Conditions Investigation

The operational conditions, including determining the amount of material used and the optimal pH for achieving the highest removal rates of COD and TSS from the paper mill effluent, were examined. These materials were used as a chemical pretreatment system.

Materials and Methods

Characteristics of the Paper Mill Effluent

The effluent used in this study was obtained from the paper mill located in the Babolkanar industrial estate in Mazandaran province. In each stage, 20 liters of effluent were collected from the kraft section outlet of the factory and stored at 4 degrees Celsius to prevent changes in its physical and chemical properties. The quality parameters of the collected effluent, such as COD, TSS, pH, TDS, and others, were determined according to the Standard Methods book.

Table1: Paper factory information

Parameter	Unit	Amount
COD	mg/L	1400.0
BOD5	mg/L	450.0
TSS	mg/L	2340.0
pH	----	6.58
TDS	mg/L	1582.0
EC	µS/cm	2320.0
Turbidity	NTU	200.0
Total Hardness	mg/L as CaCO3	324.0
Sulfate	mg/L	105.0
Nitrate	mg/L	41.0

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Chemicals Used

The chemicals used in this study to reduce pollutant parameters included aluminum sulfate [alum], ferric chloride, iron sulfate, and polyaluminum chloride [PAC]. Additionally, anionic and cationic polyelectrolytes were used as coagulant aids. To adjust the pH, sulfuric acid [H2SO4] and sodium hydroxide [NaOH] with specified concentrations were used. The concentration of all coagulants was 2%, and for coagulant aids, it was 0.1% for conducting coagulation and flocculation experiments. To prepare a 2% solution, 4 grams of the desired material was added to 200 ml of distilled water. By adding 1 ml of the prepared solution to each liter of effluent, the concentration of the used material would be 20 mg/l. To prepare a 0.1% solution, 0.2 grams of the desired material was added to 200 ml of distilled water. Thus, by adding 1 ml of the prepared solution to 1 liter of effluent, a concentration of 1000 mg/l was achieved.

Coagulation and Flocculation Process

To conduct the coagulation and flocculation experiments, a jar test apparatus with six 500 ml beakers was used. First, 300 ml of effluent was added to each container, and then various amounts of H2SO4 and NaOH were added to adjust the pH in the range of 2-9. Subsequently, different amounts of coagulants [200-1200 mg/l] and coagulant aids [1-6 mg/l] were added to the samples. For the coagulation and flocculation process, the samples were initially mixed at a high speed of 200 rpm for 2 minutes and then at a slow speed of 40 rpm for 15 minutes. After the mixing process, to settle the coagulated materials, the samples were transferred to graduated cylinders, left undisturbed for 30 minutes, and then samples were taken 2 cm from the liquid surface to examine the COD and TSS levels [25]. Additionally, at the end of each experiment, the volume of produced sludge was recorded. The experiments conducted are shown in Table 2, indicating that initially, coagulants and coagulant aids were tested at pH values of 2 to 9, and the impact of pH on the removal of COD and TSS was examined. Then, the effect of the concentration of these materials on the removal rates of COD and TSS from the effluent samples of the paper mill was investigated.

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Table2: Experiments Conducted for the Coagulation and Flocculation Process

Experiment	Coagulant	pH	Coagulant Concentration (mg/l)	Coagulant Aid	Coagulant Aid Concentration (mg/l)	Concentration (mg/l)
1	Alum	2-9	200-1200	Cationic	1-6	(4)+(1000)
2	Poly Aluminum Chloride	2-9	200-1200	Anionic	1-6	(4)+(1000)
3	Ferric Chloride	2-9	200-1200	-	-	-
4	Iron Sulfate	2-9	200-1200	-	-	-
5	-	-	-	-	-	-

3. Finding

The effluent samples collected from the paper mill were analyzed, and the results of the experiments after determining their physical and chemical properties are as follows:

Investigation of the Effect of pH on the Removal of COD and TSS

As shown in Table 1, the effluent from the paper mill contains high levels of COD and TSS. The coagulation and flocculation process was used to treat this effluent. Polymeric coagulants destabilize and settle the colloidal particles by neutralizing the forces between them. Cationic coagulants reduce the negative charge of colloidal particles by generating a positive electric charge, causing particles with opposite charges to collide and form larger particles called flocs. Usually, coagulants produce hydroxyl ions when dissolved in water. These hydroxide polymers have an amorphous structure, positive charge, and high surface area. Additionally, the formed hydroxyl ions, due to their hydrophobic properties, cause anionic organic particles to remain insoluble when in contact with their surface [26].

pH plays a crucial role in the coagulation and sedimentation processes. Changes in pH can alter the nature of organic materials and coagulants [27]. Additionally, pH is important for forming metallic hydroxides and precipitating colloidal particles. Variations in pH impact the effectiveness of coagulants in removing organic materials [27]. Generally, reducing pH in the presence of multivalent cations increases sedimentation. The impact of pH on the reduction of COD and TSS was significant, as will be discussed. All experiments were conducted at different pH values [2-9] and with a constant amount of coagulant.

As shown in Figure 1, iron sulfate caused a 12% reduction in COD at pH = 5. Polyalanine chloride and ferric chloride showed the highest COD removal at pH less than 3 [13% and

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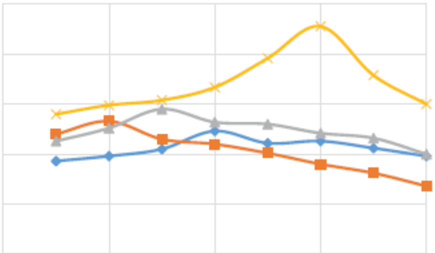
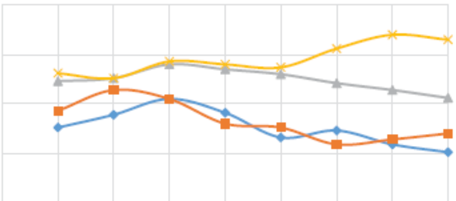
16%, respectively]. According to this chart, treating the paper mill effluent with alum coagulant resulted in a 23% reduction in COD at pH = 7.

Figure1: Effect of pH on COD Removal



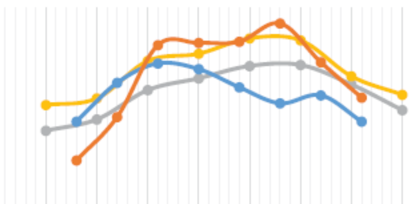
The impact of pH on TSS removal was also investigated in this study. The removal rates of TSS for iron sulfate, ferric chloride, polyalanine chloride, and alum were 45.5%, 46.4%, 49%, and 52%, respectively, as shown in Figure 2. The optimal pH for polyalanine chloride and iron sulfate was pH = 4, while for ferric chloride and alum, it was pH = 3 and 7, respectively. The difference in optimal pH may be due to severe precipitation in an acidic environment, which increases sedimentation and thus reduces TSS. Additionally, alum showed better performance in removing COD and TSS at lower concentrations than other coagulants.

Figure 2: The Effect of pH on TSS Removal



Observations indicate that the treatment of paper mill effluent is highly dependent on the initial pH of the sample. Various groups present in the effluent interact with metal cations. Carboxylic and phenolic groups collect metal cations at low pH, while hydroxyl and aliphatic hydroxyl groups interact at high pH levels. The removal of dissolved organic matter during the coagulation process at different pH values follows two specific mechanisms. At low pH, anionic organic molecules interact with metal cations to form insoluble metal complexes. At high pH and high coagulant concentrations, organic matter is absorbed by metal hydroxides, leading to sediment formation.

Figure 2: Effect of pH on COD and TSS Removal by Cationic and Anionic Coagulant Aids



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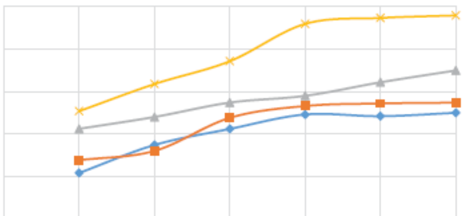
Effect of Coagulant and Coagulant Aid Concentrations on the Reduction of COD and TSS

In this study, the effect of the concentration of coagulants and flocculants on the reduction of COD and TSS was also examined. Different concentrations of coagulants in the effluent, combined with rapid mixing, created suitable coagulation conditions. Medium-speed mixing initiated the formation of flocs, followed by the adsorption of organic particles and subsequent sedimentation of insoluble solid materials, leading to the precipitation of pollutants. As shown in Figure 4, the effect of concentration on COD reduction at optimal pH was investigated. The concentration of all coagulants varied from 0.2 to 1.2 g/l. For alum, it was observed that increasing the concentration up to 0.8 g/l resulted in a COD reduction of up to 23%, with the rate of decrease becoming minimal thereafter. Therefore, the optimal dose for alum is considered to be 0.8 g/l. Additionally, the amount of sludge produced in this section was 45 ml. For polyalanine chloride and ferric chloride, rapid COD reduction was observed up to 0.8 g/l, achieving 14.5% and 13.3%, respectively. Beyond this concentration, further increases did not significantly reduce COD. Iron sulfate also resulted in a 12% reduction in COD. Based on the results obtained, it was found that alum, at very low concentrations, significantly reduces COD and can therefore be used industrially at minimal cost.

Figure 4: Effect of Coagulant Concentration on COD Removal



In this study, the effect of coagulant concentration on TSS removal at optimal pH was also investigated, and the results are shown in Figure 5. As seen in the figure, alum showed a maximum TSS reduction [57%] at 1 g/l. Ferric chloride and polyaluminum chloride reduced TSS by 47% and 51%, respectively, at 1 g/l. Beyond this concentration, increasing the coagulant amount had little effect, making 1 g/l the appropriate concentration for both coagulants. Iron sulfate significantly reduced TSS up to 0.8 g/l [45%] but was not as effective at higher concentrations.

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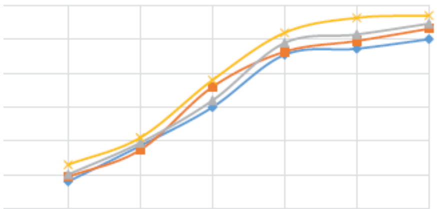
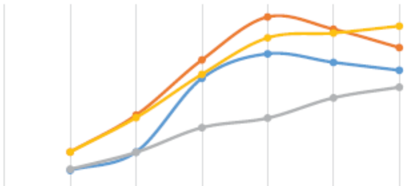
Figure 5: Effect of Coagulant Concentration on TSS Removal



The effect of the concentration of polymeric coagulant aids, both cationic and anionic, on the reduction of COD and TSS at optimal pH was also examined in this study, as shown in Figure 6. The use of anionic coagulant aid at a concentration of 0.004 g/l significantly reduced COD by 41%, while the cationic coagulant aid at the same concentration reduced COD by 37%. Moreover, as the polymer concentration increased, COD removal decreased, possibly due to the organic nature of these polymeric materials. The effect of coagulant aid concentration on TSS reduction showed that the anionic coagulant aid at an optimal concentration of 0.004 g/l reduced TSS by 56%, whereas the cationic coagulant aid, even at its highest concentration [0.006 g/l], only achieved a 42.6% reduction.

Figure 6: Effect of Polymeric Coagulant Aid Concentration on TSS and COD Removal



This indicates the higher efficiency of the anionic coagulant aid compared to the cationic one. Kaur and colleagues treated a sample of paper mill effluent on a medium scale using alum as the coagulant and chitosan [a natural polymer] as the flocculant. The maximum COD reduction at a chitosan concentration of 0.3 g/l was achieved, and further increases in chitosan amount did not improve the reduction efficiency. Additionally, a gradual increase in chitosan amount led to a reduction in TSS. Observations showed that at a concentration of 0.3 g/l of the flocculant, there was a maximum reduction of 81% TSS and 78% COD [29].

To enhance the efficiency of the chemical treatment process for paper mill effluent, the simultaneous use of coagulants and polymeric anionic and cationic coagulant aids was investigated for their effect on reducing COD and TSS. The results are shown in Figures 7 and 8. Based on the experiments conducted in the previous sections, the optimal concentration of 0.004 g/l for both anionic and cationic polymeric coagulant aids was considered, and varying concentrations of coagulants [0.2-1.2 g/l] were used. As observed,

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the use of polymeric coagulant aids significantly increased COD removal; with increasing coagulant concentration, the removal rate also increased. Alum, with a removal rate of 48.6% at a concentration of 1 g/l, was chosen as the most effective coagulant, while at the same concentration, the removal rates for polyaluminum chloride, ferric chloride, and iron sulfate were 36.2%, 38%, and 35.5%, respectively.

Figure 7: Effect of Coagulant and Coagulant Aid Combination on COD Removal

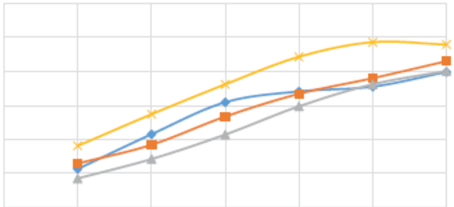
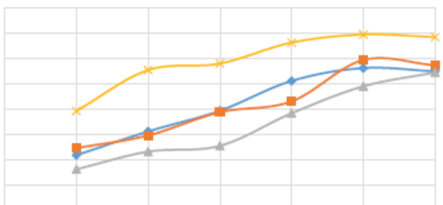


Figure 8 shows the effect of a fixed concentration of polymeric coagulant aids [0.004 g/l] and varying concentrations of coagulants [0.2-1.2 g/l] on TSS reduction. As seen, the combination of

these polymeric materials had a significant impact on TSS reduction. The optimal concentration of 1 g/l of alum not only effectively removed COD but also reduced TSS by 70%. Additionally, with increasing coagulant concentration, the TSS removal rate also increased, and the volume of settled sludge reached 38 ml. The removal rates for iron sulfate, ferric chloride, and polyalanine chloride at a concentration of 1 g/l were 56%, 60%, and 50%, respectively. Beyond this concentration, the removal rate remained almost constant. Ahmad and colleagues, in a study on the use of coagulation and flocculation methods for treating pulp and paper mill effluent, examined the efficiency of alum and polyalanine chloride [PAC] with cationic and anionic polyacrylamide. In this research, the optimal pH and concentration of alum and PAC for achieving the highest removal rates of COD and TSS were reported.

Figure 8: Effect of Coagulant and Coagulant Aid Combination on TSS Removal



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4. Conclusion

The effluent produced in the paper industry, besides being high in volume, contains significant pollution due to the high-water consumption. The treatment process is one of the most challenging and complex due to the chemical complexity of the pollutants present in the effluent. If released into the environment without treatment, it can cause severe environmental hazards. This study investigated the treatability of effluent from the paper mill located in the Babolkanar industrial estate using coagulation and flocculation processes with polymeric coagulants and charged coagulant aids. Iron sulfate, ferric chloride, polyaluminum chloride, and alum were used as polymeric coagulants, and cationic and anionic polyelectrolytes were used as coagulant aids. Based on the results, the use of coagulants and coagulant aids in treating paper mill effluent was very effective, and these findings can be used in the design and construction of a chemical treatment system for the mill.

Ethical Considerations

The authors have observed all ethical considerations, including avoiding plagiarism, duplicate publication, data fabrication, and falsification. They also declare that there are no real or material conflicts of interest that could affect the results or interpretation of the article.

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