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Removal of pharmaceutical pollutants from water by carbon nanoparticles: A kinetic investigation.

Research submitted as partial fulfillment of the requirements for the completion of the BSc Degree in Chemistry

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Acknowledgement & Dedication

Our deepest gratitude goes out to Dr. Babiker, our supervisor, for the outstanding leadership and direction he has provided. Also, we would like to express our gratitude to the chemistry professors at Imam Mohammad Ibn Saud Islamic University's College of Science for all the help and guidance they have given us. Our family's unending support and encouragement have inspired us to create this work.

Figures

الملخص باللغة العربية

يعد تلوث المياه بالمواد الصيدلانية من خلال تصريف مياه الصرف الصناعي مشكلة بيئية عالمية. ونتيجة لذلك، قدم العديد من الباحثين طرقًا مختلفة لمعالجة مياه الصرف الصحي الصيدلانية. في هذه الدراسة، تم تصنيع جزيئات الكربون النانوية من قشر القهوة (CCNPs)، وقشور الفول السوداني (PCNPs). ثم تم استخدام المواد النانوية المحضرة لإزالة السيبروفلوكساسين (CIPF) من الوسائط المائية. أظهرت النتيجة التي تم الحصول عليها أن الحد الأقصى لقيم قدرة الامتزاز لـ CCNPs وPCNPs كان 114.8 ملجم جم-1، و90.7 ملجم جم-1، على التوالي. تم الوصول لحالات الاتزان لـامتزازCIPF على PCNPs و CCNPs خلال 90 دقيقة. لقد وجد أن حركية امتزاز CIPF على CCNPs و PCNPs تتبع النموذج الحركي من الدرجة الأولى الزائفة. علاوة على ذلك، أظهرت الدراسة الخاصة بآلية التحكم في معدل الامتزاز أن نموذج الانتشار داخل الجسيمات يتحكم في امتزاز CIPF على CCNPs وPCNPs.

Abstract

Contamination of water by pharmaceutical substances through the discharge of industrial wastewater is a worldwide environmental problem. As a result, many researchers have introduced various methods for treating pharmaceutical wastewater. In this study, carbon nanoparticles were synthesized from coffee skin (CCNPs), and peanut shells (PCNPs). The prepared nanomaterials were then used to eliminate ciprofloxacin (CIPF) from aquatic media. The obtained result revealed that the maximum adsorption capacity values for CCNPs and PCNPs were 114.8 mg g-1, and 90.7 mg g-1, respectively. The equilibrium states of PCNPs and CCNPs were achieved within 90 minutes. It was found that the adsorption kinetics of CIPF on CCNPs, and PCNPs followed the pseudo-first order kinetic model. Furthermore, the investigations of the adsorption rate control mechanism showed that the intraparticle-diffusion model controlled the CIPF adsorption on CCNPs and PCNPs.

Chapter I

Introduction

1.1. Introduction

The term "water pollution" refers to any change in the quality of water, whether it be physical or chemical, that has an impact on living creatures or renders water unfit for use in vital applications. There is a significant influence on the lives of individuals, families, and communities as a whole. Due to the fact that water is essential for all living things, including people, the contamination of water poses a huge danger to the world's life [1]. Because water pollution may take many forms and have a wide range of consequences, there are many different ways to define this problem. This, in essence, is detrimental to the quality of the water since it causes disruptions to the ecology of the water and reduces its capacity to function as it should. In addition to causing harm to rivers, seas, lakes, rains, wells, and groundwater, this may also render the water unfit for human consumption, reduce its economic worth (especially in relation to fish and other aquatic life), and make it less valuable. The eventual result of water pollution is that it is unsuitable for ingestion by all living things, including people, animals, plants, and aquatic species [2]. Groundwater is essential to the river's flow and is collected through prospecting, drilling, or springs. [3]. In recent years, there has been a significant increase in water pollution, which has resulted in a shortage of water for both humans and agriculture. Since the advent of polluted water systems, outbreaks of diseases induced by water have frequently wiped out entire populations. There have been several instances of this happening.

[4]. Most of the contaminants in the ocean originate from land-based sources, such as industrial activities and waste disposal. [5]

1.2. Water contaminants and sources

1.2.1. Pesticide

Pesticides, specifically insecticides, are utilized in farming and public health to eliminate harmful insects. Unfortunately, their usage often results in environmental harm as they contaminate the soil, water, plants, and animals. This contamination can occur in various places, such as groundwater, wells, springs, rivers, lakes, water reservoirs, and ponds. As a result, it is difficult to rebalance the ecosystem after such pollution [6]. Water contamination caused by pesticides can occur in various ways. This includes accidental transfer during the spraying process from neighbouring areas, leakage from lands that use pesticides in conjunction with water movement, or direct pollution caused by using pesticides to eliminate intrusive plants in rivers. Pesticide waste is a serious problem affecting human health, causing respiratory issues, skin and eye irritations, and fatal consequences for fish and crops. This particularly affects cotton plants when irrigated with treated water and milk-producing animals when they consume contaminated water. For example, using pesticides to eliminate the Nile rose can be harmful [7].

1.2.2. Sewage

Disposing wastewater (sewage) is a major global issue because of its potential health and economic hazards. This polluted water contains harmful pollutants like organic or chemical substances (such as soap and detergents), toxic heavy metals and carbohydrates, and harmful bacteria and microbes. Wastewater contains numerous harmful bacteria that can lead to various illnesses. A gram of bodily waste (such as sweat, urine, or faeces) can contain up to 10 million viruses and one million bacteria. Some harmful bacteria include salmonella, which can cause typhoid fever and gastroenteritis; shigella, which can cause diarrheal diseases; and Escherichia coli, which can lead to vomiting, diarrhoea, and dehydration, particularly in children [8]. Additionally, Leptospira can cause inflammatory diseases in the liver, kidneys, and central nervous system, while Vibrio bacteria can cause cholera. These types of bacteria and other diseases are caused by dealing with water contaminated with sewage, whether by drinking, bathing, or even eating fish caught in these waters. Instead of living near polluted water bodies, polio, yellow fever, scabies, and malaria can be mentioned [9].

1.2.3. Chemical industry

Water pollution is caused by chemical compounds in the water that exceed natural density rates. This can happen when wastewater from mineral washing and sorting and industrial water is dumped into natural streams or sewage networks without treatment. This water may not contain heavy metals or be highly toxic, but it does contain harmful substances like chromium, carbonate, zinc, and lead that can cause skin and internal diseases. Chemical contaminants are only treated at specialized treatment plants; sewage treatment plants cannot handle them [10]. Water contaminants can enter the aquatic environment from various sources, including mines, manufacturing effluents, and light industry, as well as roof runoff in places with a metal roof. In regions where lead had not previously been exposed to gasoline, bypassed or poorly treated local effluents, road runoff containing wear layer, brake, engine materials, and lead from exhaust emissions were eliminated. Because of their high toxicity, the US Environmental Protection Agency has designated cadmium, chromium, arsenic, mercury, lead, copper, zinc, and nickel as priority control pollutants. They are on the World Food Programme's list of compounds that must be reduced or eliminated in Europe. Since 1990, cadmium, mercury, and lead emissions from all sources have consistently decreased across Europe. Fertilizers, insecticides, and mineral salts such as copper sulfate are examples of agricultural chemicals vulnerable to invading the aquatic environment [11]. Historically, animal droppings and plant materials such as manure or decaying seaweed were used to fertilize crops, followed by guano, nitrates, and phosphorus. Excessive nitrates, common in private wells, can induce "blue baby syndrome" in children allergic to nitrates [12].

1.2.4. Pharmaceutical industry

The impact of medications and their byproducts on the environment is significant. These drugs can positively and negatively affect various environmental factors, including biota. Through advanced scientific methods, it has been discovered that several pharmaceutical compounds exist in high concentrations in surface water, subsurface water, groundwater, residential wastewater, municipal wastewater, and industrial effluents[13]. To support pharmaceutical manufacturers, many countries have established excise duty-free zones, which has resulted in the production of a wide range of pharmaceutical items and complex, non-biodegradable toxic waste byproducts. Unfortunately, some waste is released into the environment without adequate treatment [13].

1.3. Water treatments

Public and wastewater systems utilize various water treatment techniques to ensure clean and safe drinking water for their communities [14].

1.3.1. Heating

One of the easiest and quickest ways to purify water is by boiling it. Water from various sources and delivery channels may contain harmful substances, such as parasites and germs, which cannot be seen with the naked eye but can be deadly. To purify water through boiling, it must be brought to a boil and kept at that temperature for 1-3 minutes. People living in high-altitude areas should boil their water longer than those in lower elevations, as water boils at lower temperatures at higher altitudes. After boiling, the water should be covered and allowed to cool before consumption. For well water, it is advisable to wait for chemicals to settle before filtering out pure water for use [15].

1.3.2. Filtration

Filtration is one of the most effective methods of purifying water, and when done correctly, it helps remove water from contaminants. This technology purifies water and makes it safe for human consumption using chemical and physical processes. With a simple and quick procedure, filtration eliminates major chemicals and small, harmful pollutants that cause diseases. Because filtration does not eliminate all mineral salts, filtered water is healthier than water treated in other ways[16]. It is one of the most effective water purification procedures because it uses a chemical absorption mechanism to effectively remove undesirable substances from water. Compared to reverse osmosis, filtration is thought to be more successful in selectively removing considerably smaller molecular pollutants like chlorine and pesticides. Another element that makes filtration less expensive is requiring less energy than distillation and reverse osmosis. It is a cost-effective water purification method because minimal water is lost during filtration [17].

1.3.3.Reverse osmosis

Reverse osmosis is a method that uses a partially porous membrane to eradicate undesirable particles, including ions and larger compounds, from drinking water. This technique effectively removes dissolved and suspended chemical and biological species such as bacteria. Reverse osmosis is widely used in both industrial settings and for water purification. The process involves pushing water across a semi-permeable membrane to filter out impurities. Reverse osmosis produces clean water ideal for various applications, including drinking water systems, industrial boilers, food and beverage processing, cosmetics, pharmaceutical production, and seawater desalination [18].

1.3.4.Ozonation

Water purification through ozonation involves injecting ozone into the water, a highly effective oxidant. Ozone is a gas composed of three oxygen atoms (O_3), and it generates highly reactive oxygen species that can attack a wide range of chemical substances and microorganisms. Ozone treatment is an advanced oxidation process that can effectively purify water and break down organic and inorganic contaminants. To create ozone, oxygen (O_2) is exposed to high electric voltage or UV radiation, which consumes a significant amount of energy and is expensive. Nonetheless, the necessary amount of ozone can be produced on-site for various water treatment applications [19].

1.3.5. Ion Exchange

The ion exchange (IE) method has been a popular chemical and environmental engineering approach for several years. Initially, it was primarily employed for water softening, which entails the removal of calcium and magnesium ions from water at the treatment plant or as a point-of-use treatment method. Further, it was employed to produce completely demineralized water for industrial applications. However, with new regulations on certain inorganic chemicals, the IE method is being applied in new ways for water treatment. Pollutants such as nitrate, arsenic, selenium, barium, radium, lead, fluoride, and chromate are among the most likely targets for elimination utilizing [20].

1.3.6. Chemical precipitation

Chemical precipitation is widely regarded as the most efficient method for removing heavy metals from wastewater. It is commonly used in industries since it is affordable and simple to set up. Chemical precipitation, also known as reagent coagulation, is a method of removing contaminants from purified water by changing the pH, electro-oxidizing potential, or coprecipitation with precipitating agents (coagulants) such as ferrous or aluminium sulfates. Reagent oxidation is a subset of reagent coagulation in which oxidizing reagents (for example, potassium permanganate or bichromate) are added to a purified solution to eliminate organic contaminants or change the valence of multivalent ions after precipitation. Electrocoagulation is a contaminant precipitation method involving transferring ions from a dissolving anode into purified water using an electric current [21]. Iron or aluminium are common anode materials for electrocoagulation. When an electric current is transmitted between electrodes in a solution containing chloride ions, hypochlorites develop on anodes, causing organic contaminants to decompose. There are two common chemical precipitation techniques: hydroxide precipitation and sulfide precipitation. Among the two, hydroxide precipitation is more commonly used because it is cost-effective, simple, and easy to regulate the pH. The solubility of metal hydroxides is covered within the pH range of 8.0-11.0. Flocculation and sedimentation can also quickly remove metal hydroxides [22]. Lime is the most widely used base in industrial settings because it is cheap and easy to handle. In a recent study by Mirbagheri and Hosseini, Ca(OH)2 and NaOH were used to get rid of Cu(II) and Cr(VI) ions from wastewater. The use of ferrous sulfate converted Cr(VI) to Cr(III), and the maximum precipitation of Cr(III) occurred at pH 8.7 with the introduction of Ca(OH)2. The content of chromate decreased from 30 to 0.01 mg/L. However, hydroxide precipitation has some drawbacks since it produces a large amount of low-density sludge that causes dewatering and disposal issues [23].

1.3.7. Adsorption

The process of transferring substances between two phases, such as a liquid and a solid, a gas and a liquid, or a gas and a solid, is known as adsorption. Contaminants can be removed from wastewater through intermolecular forces. There are two types of adsorption: physisorption and chemisorption. Physisorption occurs when there are weak physical forces, such as van der Waals forces, and the process is reversible. This adsorption type occurs when the adsorbate's critical temperature is reached or is close to it. In contrast, chemisorption involves forming a chemical bond between the adsorbate and the solid surface. However, it only occurs as a monolayer, and the adsorbates are typically washed away. Both procedures can occur simultaneously or depending on the situation. When investigating adsorption processes, factors such as the surface area, type and starting concentration of adsorbate, solution pH, temperature, interfering chemicals, and nature and dose of adsorbent should be considered [24, 25].

1.4. literature review

Pharmaceutical wastewater is distinguished by high antibiotic content, significant biological toxic effects, and a wide range of water volume and concentration fluctuations. Ciprofloxacin (CIPF) is an antibiotic belonging to the fluoroquinolone class. It treats bacterial infections such as bone and joint infections, intra-abdominal infections, respiratory tract infections, infectious diarrhoea, skin infections, typhoid fever, and urinary tract infections. It can be used in combination with other antibiotics to treat different illnesses. The medication can be administered orally or intravenously with eye or ear drops. CIPF, an antibiotic utilized in human medicine and livestock farming, is being released into bodies of water such as streams and lakes. In recent years, the concentration of this chemical has increased, and its tough chemical structure makes it difficult to remove using conventional methods. The residual CIPF in the environment has become a new micropollutant that promotes the formation of bacterial resistance genes and threatens the balance of ecosystems and human health [26]. Numerous oxidation methods have been recently researched and developed to treat Fluoroquinolones. These include chlorine oxidation, ozone oxidation, hydrogen peroxide oxidation with ultraviolet light, photocatalytic oxidation, permanganate oxidation, and ultrasonic degradation. CIPF concentrations in water typically range from ng/L to g/L, although some instances of mg/L have unfortunately been discovered. The concentration of CIPF tends to increase gradually over time. However, CIPF has a significant chemical interference and is toxic to microorganisms, making it difficult for them to absorb and utilize. As a result, materials like MOFs, synthetic chitosan, or activated carbon are often used for CIPF adsorption, but this technique alone cannot completely neutralize and degrade CIPF. Advanced oxidation techniques are therefore recommended to break down and eliminate it. In China, more than 5340 t of CIPF was reportedly used in 2013, making it the fluoroquinolone antibiotic with the second-highest utilization rate. However, regular use of CIPF can lead to pathogenic bacteria becoming resistant to medication, which poses a risk to human health if they persist in the environment for an extended period [26, 27]. Additionally, CIPF can disrupt the regular release of hormones, leading to mental health issues and impairing normal metabolism. CIPF wastewater must be treated using highly effective techniques to avoid these negative effects.CIPF concentrations in water typically range from ng/L to g/L, although some instances of mg/L have unfortunately been discovered. The concentration of CIPF tends to increase gradually over time. However, CIPF has a significant chemical interference and is toxic to microorganisms, making it difficult for them to absorb and utilize. As a result, materials like MOFs, synthetic chitosan, or activated carbon are often used for CIPF adsorption, but this technique alone cannot completely neutralize and degrade CIPF [28].

1.5. Aim of the study

This study aims to prepare carbo nanoparticles from agricultural waste as low-cost sorbents. The efficiency of the prepared nanomaterials will be studied for eliminating pharmaceutical contaminants from water. The CIPF drug will be used as a model water pharmaceutical pollutant. The kinetic of CIPF removal will be investigated, including the adsorption rate-order and the adsorption rate-control-mechanism.

Chapter II

Experimental

2. Materials and methods

2.1. Materials

CIPF was supplied from LOBA-CHEM, India. Carbon nanoparticles were synthesized from coffee skin (CCNPs) and peanut shells (PCNPs).

2.2. Adsorption of CIPF

A stock solution of CIPF (100 ppm) was prepared by dissolving 0.1 grams of CIPF in 1.0 L of distilled water. In a 100 mL beaker, a mass of 50 mg was measured for each sorbent. A volume of 100 mL of the CIPF solution was added to each beaker, and the mixture was stirred using a magnetic stirrer. Afterwards, 10 mL of each mixture was extracted periodically until equilibrium. In order to investigate the impact of contact time on the adsorptions of CIPF onto CCNPs and PCNPs at various time intervals, a sample was taken, filtered with a filter syringe, and measured with a UV-Vis spectrophotometer.

Chapter III

Results and Discusion

3. Results and discussion

3.1 Adsorption of CIPF

The experimental adsorption isotherms are frequently derived using an adsorbent's adsorption capacity, which is determined by the mass balance on the sorbate in a system with solution volume. Equ. 1 was used to determine the adsorption capabilities of each adsorbent for CIPF at equilibrium under the experimental conditions. Fig. 1 illustrates the impact of contact time on CIPF adsorption by CCNPs and peanut shells PCNPs. The equilibrium states of CIPF removals by PCNPs and CCNPs were achieved within 90 min, with maximum q_t values of 114.8 and 90.7 mg/g, respectively. Therefore, using CCNP shows great promise in effectively removing pharmaceutical waste due to its exceptional adsorption abilities.

$$q_t = \frac{(C_o - C_t)\,V}{m}, \qquad (1)$$

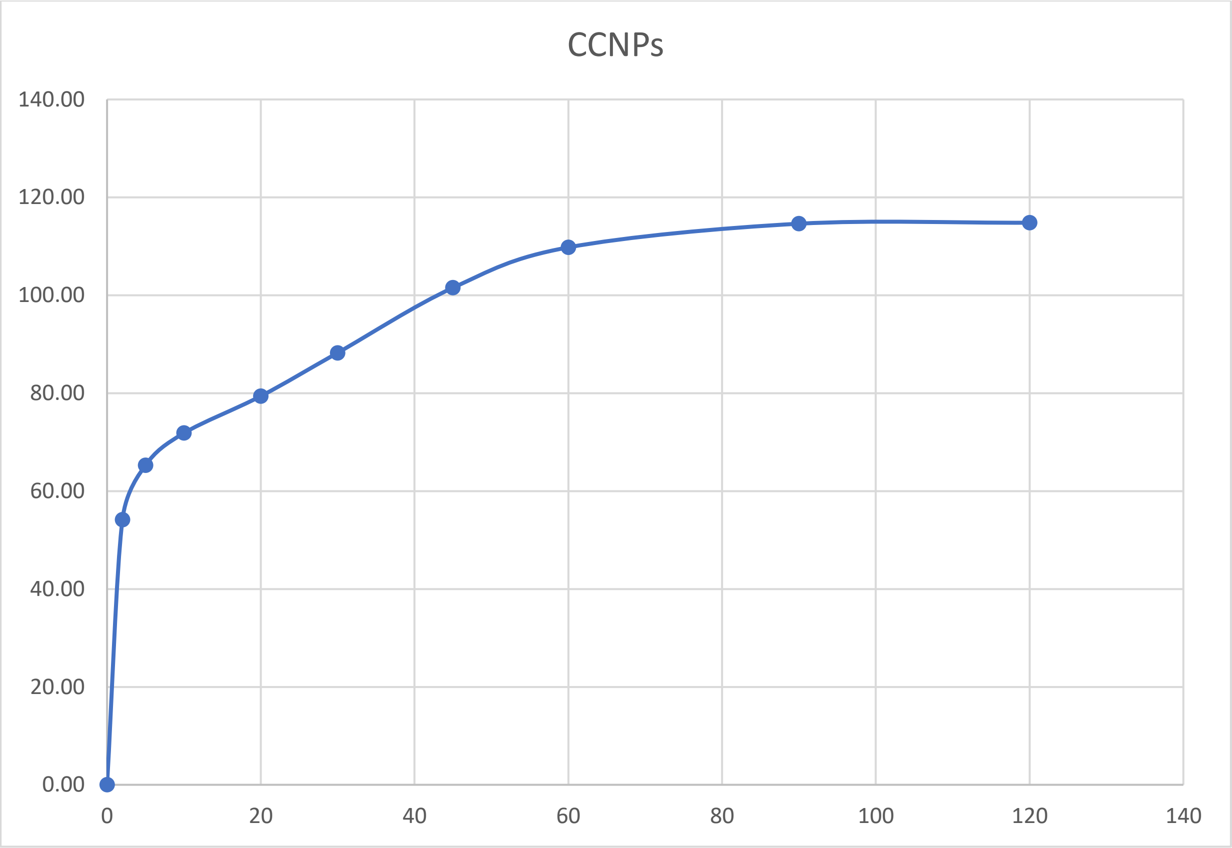

Fig1: Effect of contact time on CIPF adsorption by CCNPs**.**

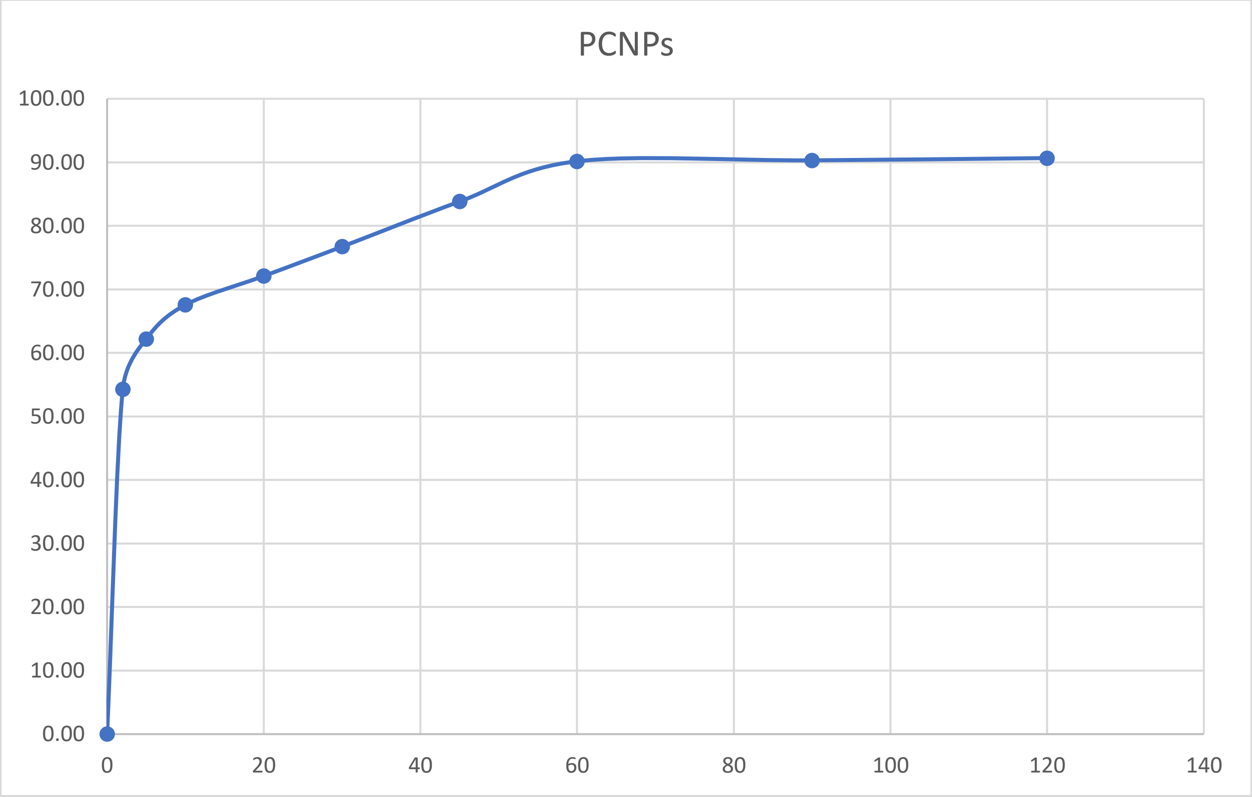

Fig2: Effect of contact time on CIPF adsorption by PCNPs**.**

Table1: Equilibrium time and Maximum adoption capacity of CCNPs, and PCNPs sorbents.

Sorbents	Equilibrium time	Q_t (mg/g)
CCNP	90	114.8
PCNP	90	90.7

3.2. Adsorption Rate order Study

Adsorption kinetics provides insight into the reaction rate and the sorption mechanism [29]. The pseudo-first-order (PFO) and pseudo-second-order (PSO) kinetic models were employed to examine the adsorption rate of CCNPs and PCNPs sorbents. The two models are described correspondingly by formulae 2 and 3.

$$ln(q_e - q_t) = ln\, q_e - k_1.t \qquad (2)$$

$$\frac{1}{q_t} = \frac{1}{k_2.q_e^2\, t} + \frac{1}{q_e} \qquad (3)$$

Where qe (mg g1) is the adsorption capacity at equilibrium and m, v, Ct, and C o are the mass of the sorbent in grams, the volume of the solution in mL, and th e concentration of the solution in mg/L at time t (min), respectively.

The constants describing PFO and PSO are k1 (min-1) and k2 (g mg-1 min-1) respectively. Linear plots of PFO results for the adsorption of CIPF on CCNPs and PCNPs are presented in Fig. 3 and 4, while the PSO plots for removing CIPF onto CCNPs and PCNPs are displayed in Fig. 5 and 6, respectively. Table 1 contains the obtained results for the PFO and PSO models. The second-order kinetic model's correlation coefficients (R^2) are near to 1. Therefore, the kinetics of CIPF adsorption onto the CCNPs, and PCNPs adsorbents can be described well by the PSO equation.

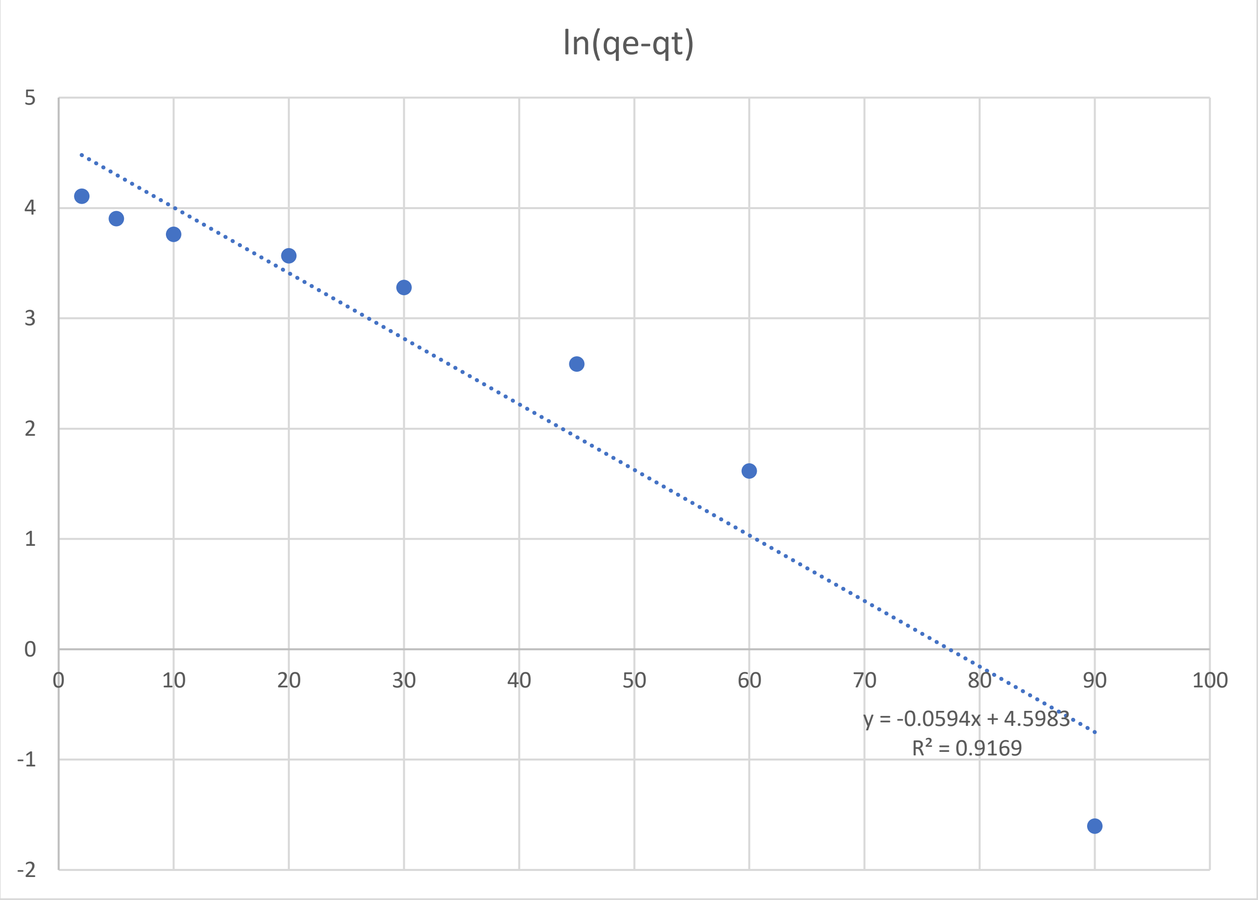

Fig 3: Plot of PFO for CIPF adsorption by CCNPs

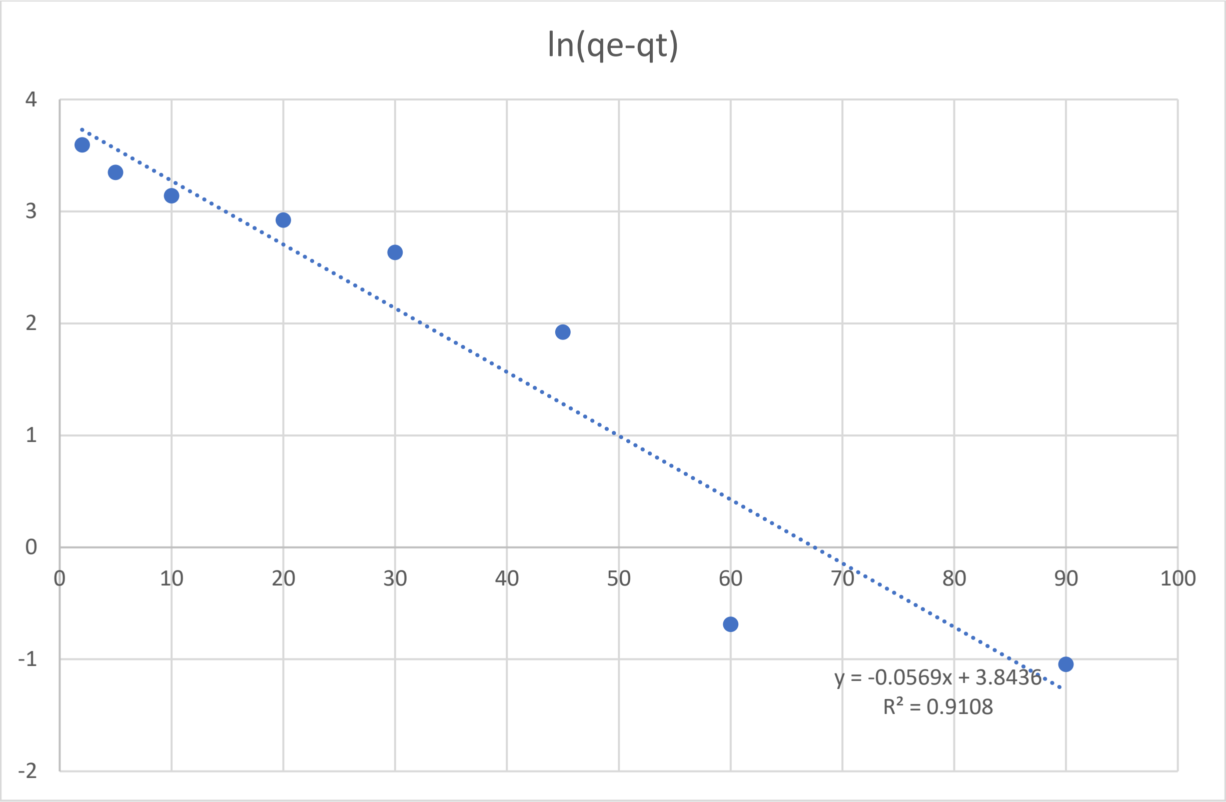

Fig 4: Plot of PFO for CIPF adsorption by PCNPs

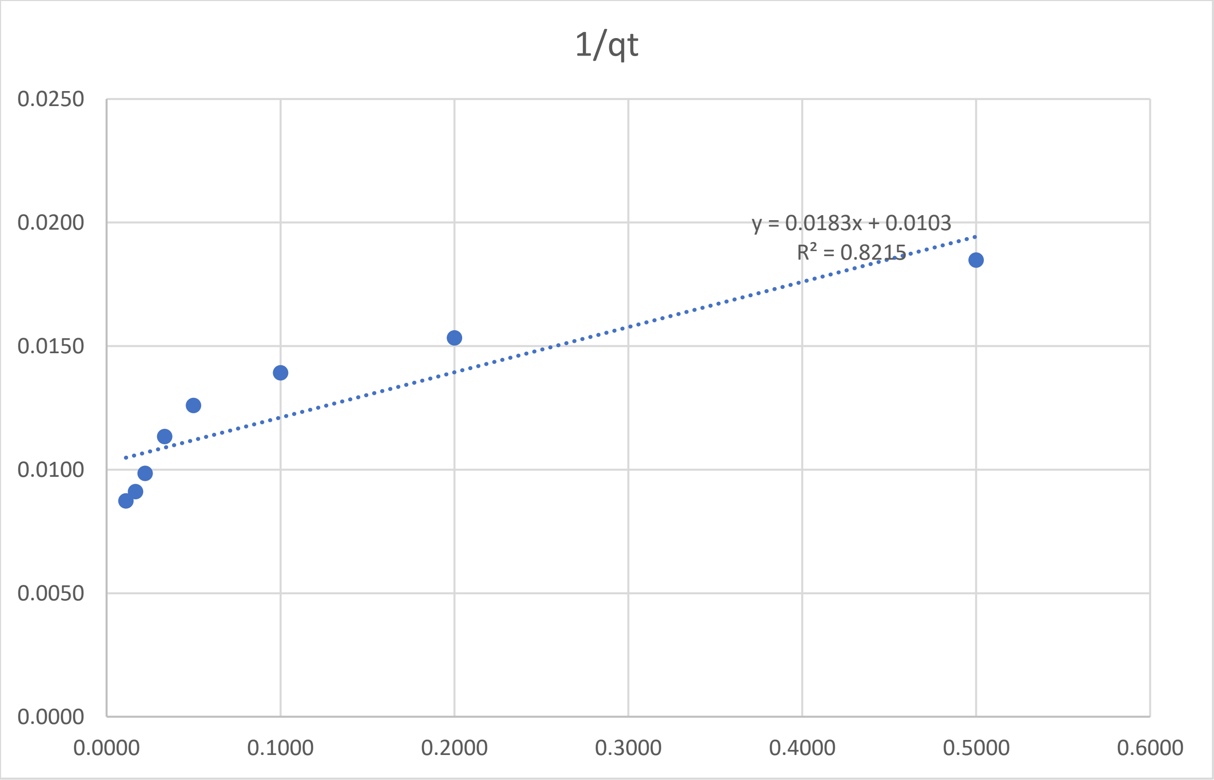

Fig 5: Plot of PFO for CIPF adsorption by CCNPs**.**

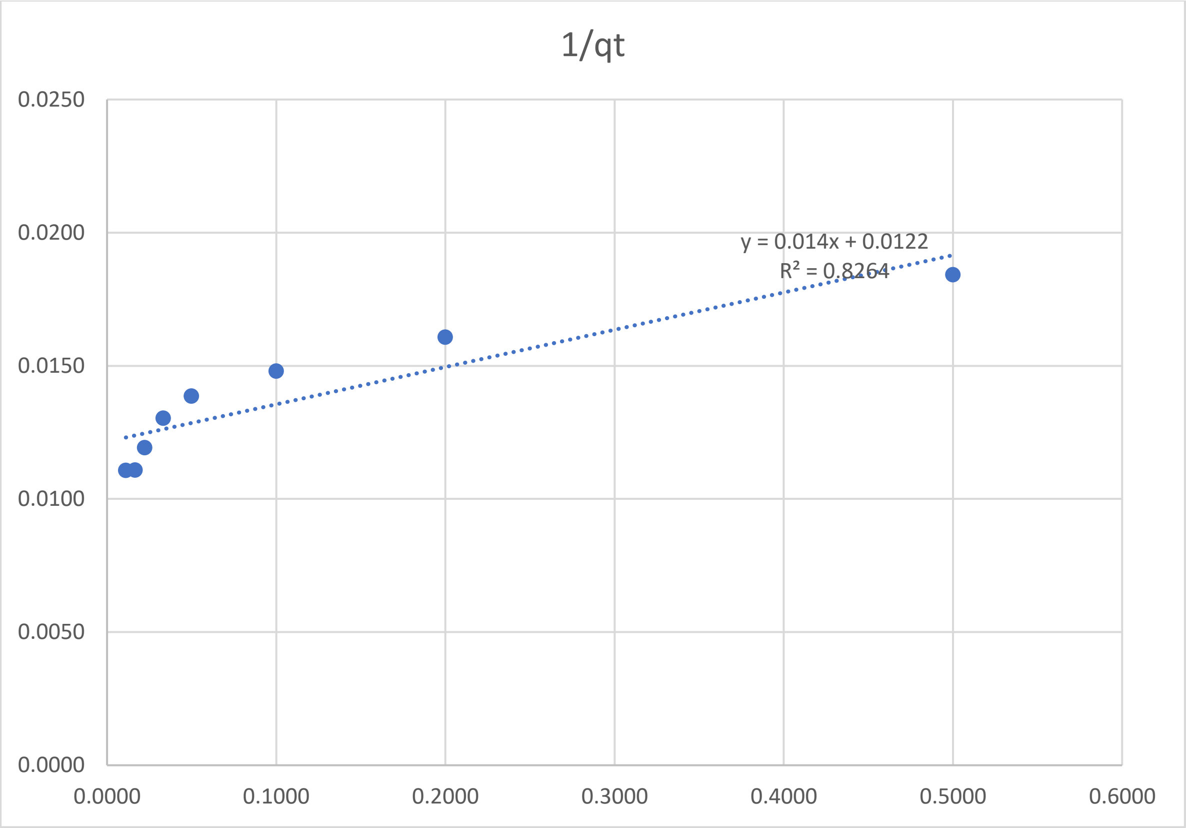

Fig 6: Plot of PSO for CIPF adsorption by PCNPs.

Table 2: The adsorption rate order results for eliminating CIPF using CCNP, OCNP, and PCNP.

adsorbent	q_e exp. (mg g^{-1})	*PFO*			*PSO*		
		qe cal. (mg g^{-1})	R^2	k_1	q_e cal. (mg g^{-1})	R^2	k_2
CCNPs	114.8	69.97	0911	0.0632	124.92	0.822	0.00409
PCNPs	90.7	33.33	0.908	0.0697	87.44	0.826	0.000002

3.3 Adsorption control mechanism

The adsorption process occurs in two parts. Firstly, adsorbate molecules are transferred from the liquid to the surface of the solid sorbent. Secondly, the molecules pass through the solid sorbent and into the inner layers. The rate control mechanism, also known as the slowest sorption step, regulates the adsorption rate. A study was conducted to investigate the rate-control mechanism for CIPF adsorption on CCNPs and PCNPs. The liquid-film-diffusion model (LFD, Eq. 4) and the intraparticle-diffusion model (IPD, Eq. 5) were used for this purpose [30, 31].

$$q_t = K_{IP} * t^{\frac{1}{2}} + C_i \tag{4}$$

$$\ln(1 - F) = -K_{LF} * t \tag{5}$$

Where: k_{ip} (mg g^{-1} $min^{-1/2}$) and k_{LF} (min^{-1}) are the IPDM, and the LFDM constants, respectively. C_i (mg g^{-1}) boundary layer factor. The linear plot for the CIPF adsorption on CCNPs and PCNPs via LFDM were monitored in Fig. 7 and 8 respectively; while the IPDM investigation was monitored in Fig 9 and 10. The computed results for LFDM and IPDM were gathered in Table 3. The rate control mechanism obtained revealed that IPDM controlled the CIPF adsorption on the CCNPs and PCNPs.

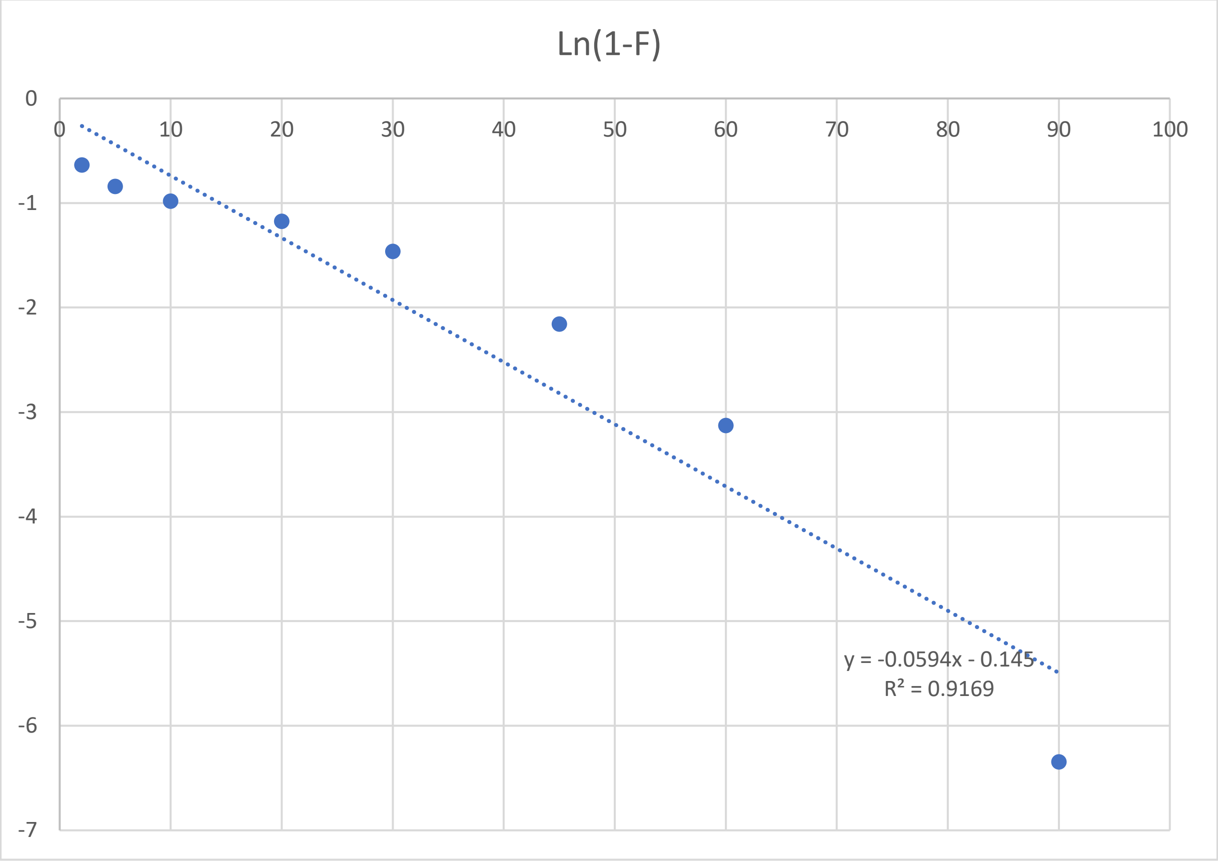

Fig 7: LFDM for CIPF adsorption by CCNPs.

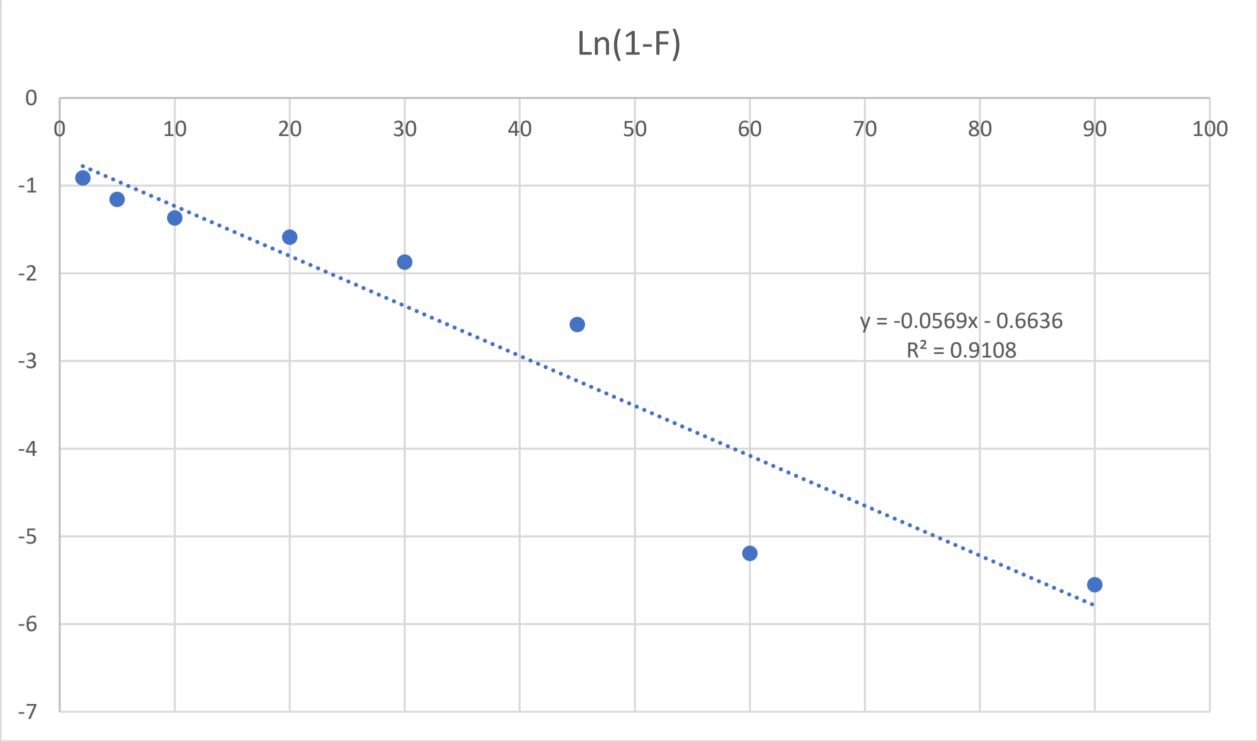

Fig 8: LFDM for CIPF adsorption by PCNPs.

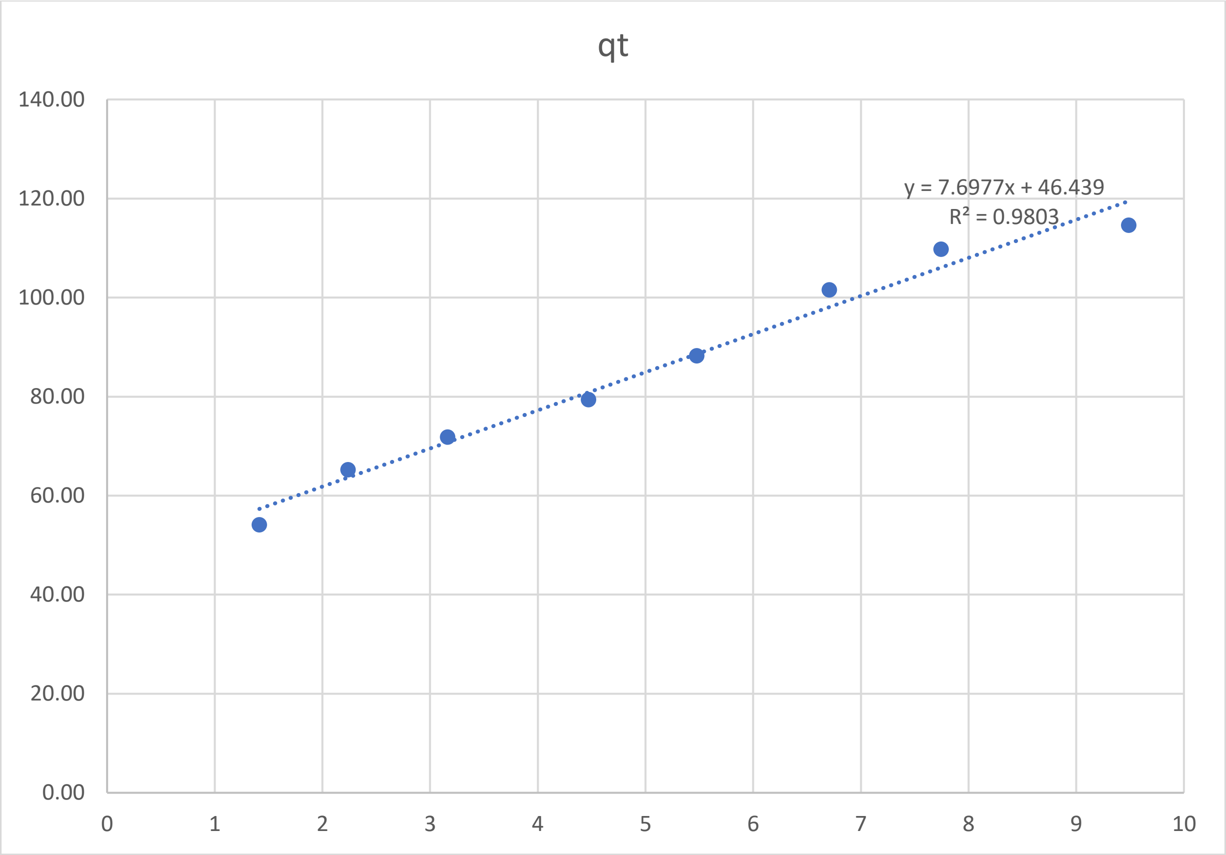

Fig 9: IPDM for CIPF adsorption by CCNPs.

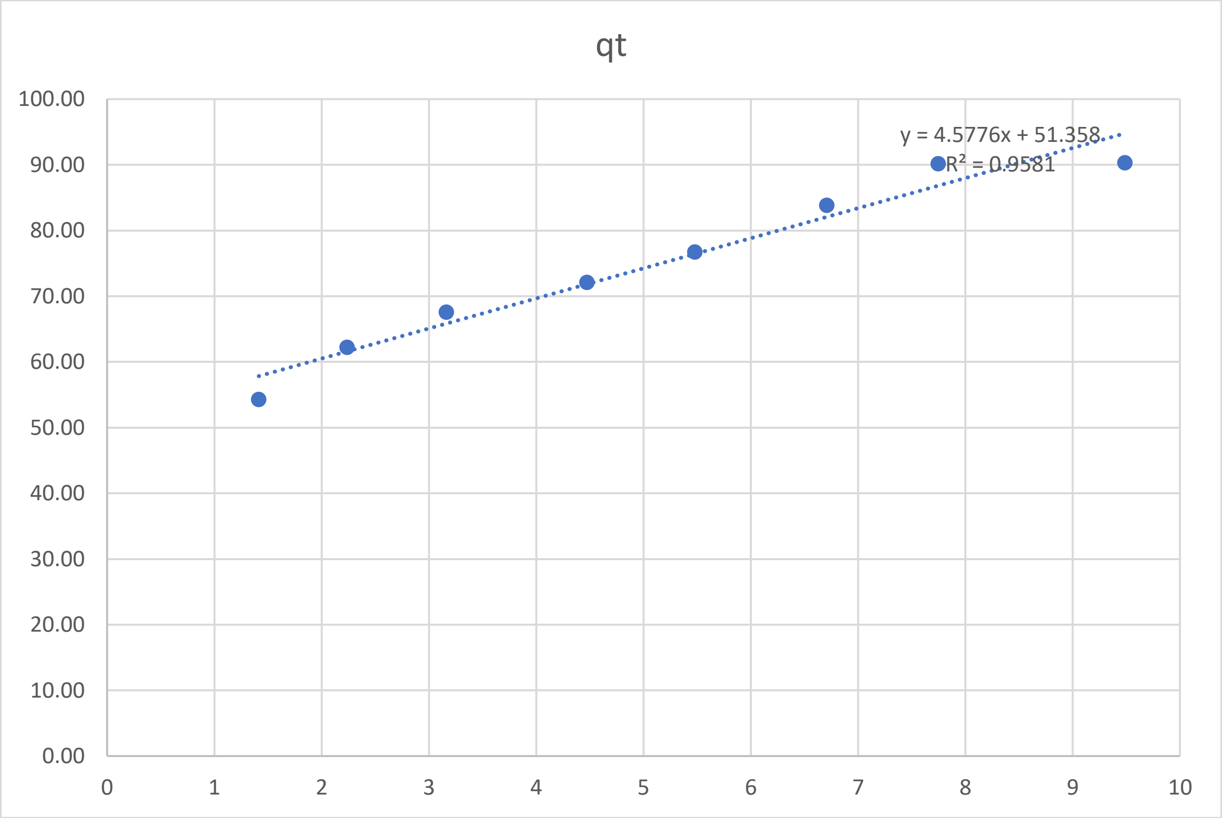

Fig 10: IPDM for CIPF adsorption by PCNPs.

Table 3: The mechanism controlled the adsorption process

Sorbent	*LFDM*		*IPDM*	
	K_{LF} (min^{-1})	R^2	K_{IP} (mg g^{-1} min$^{0.5}$)	R^2
GCNP	0.0697	0.917	15.82	0.980
GCNP	0.0751	0.911	13.70	0.958

3.4. Conclusion

The CCNP, OCNP and PCNP were employed to remove CIPF from water. The adsorption of the CIPF on PCNPs and CCNPs required 90 min to reach equilibrium. The maximum adsorption capacity values for CCNPs and PCNPs sorbents were found to be 114.8 mg g-1, and 90.7 mg g-1, respectively. Based on the study results, it was found that the adsorption kinetics of CIPF on CCNP, and PCNP followed the PSO. Furthermore, the adsorption rate control mechanism investigations revealed that IPDM controlled the CIPF adsorption on CCNP, and PCNP.

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