

# Synthesis and Application of Cellulose–Iron Nanomembrane for Removal of Pharmaceutical Contaminants Using Photocatalytic UV Reactor

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## Abstract

Pharmaceutical contaminants in the water pose serious environmental and health risks due to their persistence and resistance to conventional treatment methods. This study developed for a sustainable cellulose–iron nanomembrane integrated with a UV-assisted photocatalytic reactor for removal of amoxicillin from aqueous solutions. Green synthesis of iron oxide (Fe<sub>2</sub>O<sub>3</sub>) nanoparticle using *Punica granatum* (pomegranate) peel extract was carried out using it as a natural reducing agent and stabilizer. The prepared iron oxide nanoparticles were incorporated into the cellulose acetate matrix and fabricated into a nanofibrous membrane via electrospinning technique and further treated using citric acid cross-linking to ensure better stability. The synthesized nanoparticles and membranes were characterized by Particle Size Analysis (PSA), Fourier Transform Infrared Spectroscopy (FTIR), UV–Visible spectroscopy, and Thermogravimetric Analysis (TGA). Experimental results illustrate successful synthesis and fabrication of FeO nanoparticles with an average particle size of 225.4nm and an enhanced thermal stability. Photocatalytic degradation tests demonstrated the effective removal of amoxicillin under UV radiation with 90% removal achieved in 60minutes with cellulose-iron nanomembrane. The removal is attributed to the synergistic action of adsorption by nanomembrane and photo-oxidation generated by reactive oxygen species due to activated FeO

NPs upon UV exposure. This cellulose-iron nanomembrane shows to be a highly effective and environmentally friendly material for treating pharmaceutical wastewater and advancing water purification technology.

**Keywords:** Green Synthesis, Iron Oxide Nanoparticles,  $\text{Fe}_2\text{O}_3$ , Cellulose Acetate Nanomembrane, Electrospinning, Photocatalytic Degradation, Amoxicillin Removal, UV-Assisted Photocatalysis, Pharmaceutical Wastewater Treatment, Water Purification.

## 1. Introduction

Pharmaceutical pollutant contaminated in water has been a global environmental concern, as it is readily available in the aqueous medium. Antibiotics are ubiquitously found in the water bodies and sources due to their ceaseless disposal from hospitals, household activities, pharmaceutical industries and agricultural activities, leading to its recalcitrance and consequently forming threats on both the environment and human beings by increasing the rate of antibiotic resistance [1]. The amoxicillin is the most used antibiotic and its presence is prevalent in wastewater owing to its incomplete metabolism by the human body and persistent discharge into aquatic system thereby posing a severe need for an effective and green technology for pharmaceutical wastewater treatment [2]. Though conventional techniques like coagulation, biological treatment and adsorption provide partial removal of pharmaceuticals, nanomaterial based and membrane integrated techniques are being of great interest due to their larger surface area, enhanced reactivity and potential to support advanced oxidation process [2] [3].

The iron oxide nanoparticle ( $\text{Fe}_2\text{O}_3$  NPs) is an attractive material for environmental remediation due to its catalytic activity, stability, non-toxicity and cost-effectiveness. However, traditional methods for iron oxide NPs synthesis require hazardous chemical reagents. Therefore, the green synthesis of iron oxide NPs using plant extracts provides a clean and safe route in which phytochemicals present in the plant acts as reducing and stabilizing agents in the nanoparticle formation process [4]. The structural, morphological and optical property plays a significant role in the overall performance of a nanocomposite membrane for water treatment applications [25], [34], [35]. The rich composition of phenols and antioxidants in *Punica granatum* (pomegranate) peel makes it appropriate for green synthesis of iron oxide NPs which leads to significant reduction of chemical wastage [5] [6]. The results published in previous works clearly indicate that green-synthesized iron oxide nanoparticles can perform

efficiently for water treatment application with respect to their stability and environmental friendliness [19], [24], [30], [31].

Cellulose acetate (CA) is a bio-compatible and bio-degradable membrane material widely used due to the facility of fabricating electrospun membrane. The incorporation of iron oxide nanoparticle in the CA membrane could enhance the adsorption and photo-catalytic activity. The electrospinning is a suitable technique for the development of nanofibrous membrane to treat wastewater [7] [8]. Few studies have focused on the combined role of green-synthesized iron oxide NPs using pomegranate peel extract, CA, electrospinning and UV-assisted photo-catalytic reactor for the treatment of amoxicillin, where adsorption mechanism of the developed membrane and photo-catalytic oxidation performance was explored in a combined approach for a rapid removal. In this work, we describe the synthesis of green-synthesized Fe<sub>2</sub>O<sub>3</sub> NPs using P. Granatum peel extract and subsequent incorporation into electrospun cellulose acetate membranes for efficient photocatalytic degradation of amoxicillin under UV irradiation. The developed system integrated adsorption, membrane separation, and photocatalytic oxidation to achieve improved efficiency. Electrospun cellulose acetate membranes, due to their high porosity, large surface area and tunable functionalization, have been an attractive option for environmental remediation [15], [16], [29].

## **2. Materials and Methods**

### **2.1 Preparation of Punica Granatum (Pomegranate) Peel**

Punica granatum (pomegranate) peels from fresh juice shops at GCT Coimbatore was collected, which contains large number of phenolic compounds and antioxidants beneficial to the nature. Initially, these peels were washed vigorously using disinfected water followed by drying, grinding to the finest powder form. To extract the required compounds from the peel powder, 5 g of pomegranate peel powder was mixed with 100 ml of disinfected water and boiled for 15 min so as to enhance the solubility of the desired components. After cooling, filtered the solution using Whatman filter paper to remove the solid residue [6]. The clear filtrate was collected and was used as the natural reducing agent as well as stabilizing agent in the following synthesis steps [6].

## 2.2 Synthesis and Purification of Iron Oxide Nanoparticles

Fe<sub>2</sub>O<sub>3</sub> nanoparticles were synthesized by using FeCl<sub>3</sub> solution and pomegranate peel extract via a green synthesis process [7]. A 0.05 M FeCl<sub>3</sub> solution was gradually added to pomegranate peel extract while stirring continuously at room temperature. The reduction of Fe ions to FeO NPs was confirmed with the color changes from yellow to dark black and was further stirred to produce the product. The obtained nanoparticles were washed several times with distilled water and ethanol to remove unreacted compounds, and followed by drying using hot air oven before membrane fabrication.

## 2.3 Fabrication of Cellulose–Iron Nano Membrane

Cellulose acetate-iron nanoparticle spinning solution, 4g of cellulose acetate were dissolved in 10ml acetone. Later 0.3 g of Fe<sub>2</sub>O<sub>3</sub> nanoparticles was dispersed in 30ml acetone and was treated with ultrasonic for 30 min to achieve uniform dispersion [8]. Then it was poured into cellulose acetate solution and stirred continuously for 40 min until a homogeneous solution was formed. The prepared spinning solution was electrospun into nanofibrous membrane at an applied voltage of 14 kV, flow rate of 0.5 mL h<sup>-1</sup>, and tip to collector distance of 15 cm. The prepared membrane was then cross-linked by immersion in citric acid solution, followed by thermal treatment at 120°C for 1 hour. This would strengthen the membrane due to the formation of ester bonds within the cellulose acetate membrane and provide a higher stability [9] [10]. It has been reported that the nanofillers and nanocomposites incorporated into membranes show improved membrane performance in term of adsorption capacity and pollutant removal efficiency [14], [22], [23], [33].

## 2.4 Characterization of Nanoparticle Analysis

The synthesized Fe<sub>2</sub>O<sub>3</sub> NPs was characterized using particle size analyzer (PSA/DLS) and Fourier transform infrared spectroscopy (FTIR). The average particle size, size distribution and polydispersity index of the FeO NPs was obtained using PSA [11]. The functional group, formation of Fe-O bonds and presence of chemical functional groups derived from the bioactive compounds from the pomegranate peel extract and that incorporated on the nanoparticle was investigated using FTIR [12].

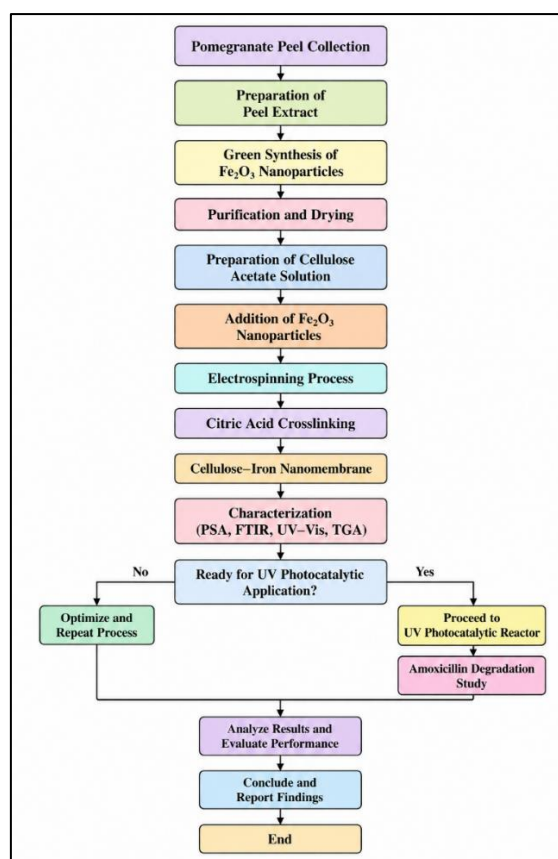
## 2.5 Characterization of Cellulose Acetate- Iron Nano Membrane

The prepared cellulose acetate-iron nanomembrane was characterized by using different characterization techniques to assess its structural, chemical, optical and thermal properties. Functional group analysis of the membranes were determined using FTIR so that the Fe<sub>2</sub>O<sub>3</sub> nanoparticles could be confirmed to be successfully incorporated into the cellulose matrix [12]. The optical properties of the membrane were assessed using the UV-Visible spectrophotometer and nanoparticle incorporation could also be verified. TGA was performed on the developed membrane in order to investigate thermal stability of the material and its thermal degradation profile [17].

**Table 1.** Electrospinning Parameters Used for Membrane Fabrication

Parameter	Value
Cellulose acetate	4 g
Fe <sub>2</sub> O <sub>3</sub> nanoparticles	0.3 g
Solvent	Acetone
Voltage	14 kV
Flow rate	0.5 mL h <sup>-1</sup>
Tip-to-collector distance	15 cm
Crosslinking temperature	120°C

Table 1 shows the experimental parameters of the synthesis of the nanomembrane. All experimental parameters were optimized for obtaining a uniform, continuous and non-defect fibrous network containing uniformly distributed nanoparticles. Moreover, thermal crosslinking was used so as to enhance the thermal and mechanical stability of the developed nanomembrane which can effectively be used for the photocatalytic degradation of wastewater.



**Figure 1.** Experimental Workflow

### 3. Characterization of Iron Nano Particle and Nano Membrane

The overall experimental methodology adopted in this study is illustrated in Figure 1. The workflow includes pomegranate peel extract preparation, green synthesis of the  $\text{Fe}_2\text{O}_3$  nanoparticles, fabrication of cellulose–iron nanomembrane through electrospinning and citric acid crosslinking, material characterization, and evaluation of amoxicillin degradation in a UV photocatalytic reactor.

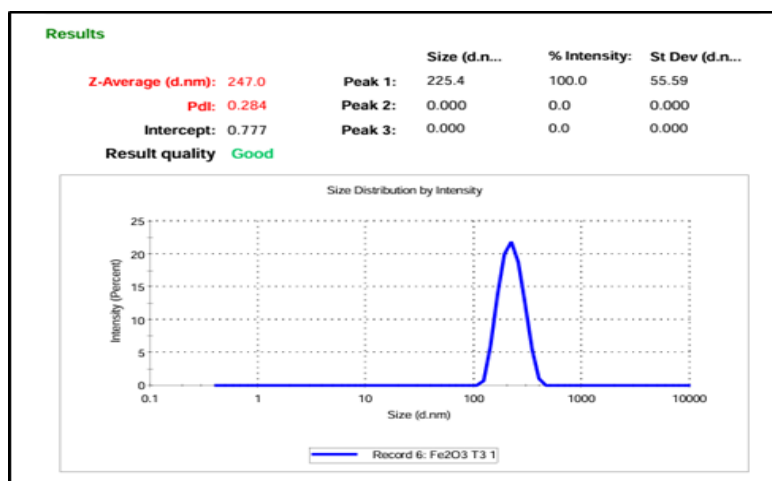
#### 3.1 Characterization of Iron Nanoparticle

The synthesized  $\text{Fe}_2\text{O}_3$  nanoparticles were characterized for Particle size distribution, Average particle size and uniform particle size of nanoparticles by PSA and for the presence of functional groups related to nanoparticles synthesis and stability by FTIR.

##### 3.1.1 Particle Size Analyzer (PSA)

The particle size distribution of the synthesized  $\text{Fe}_2\text{O}_3$  nanoparticles was measured using the Particle Size Analyzer (PSA) and is as shown in Figure 2. The Z-average particle size

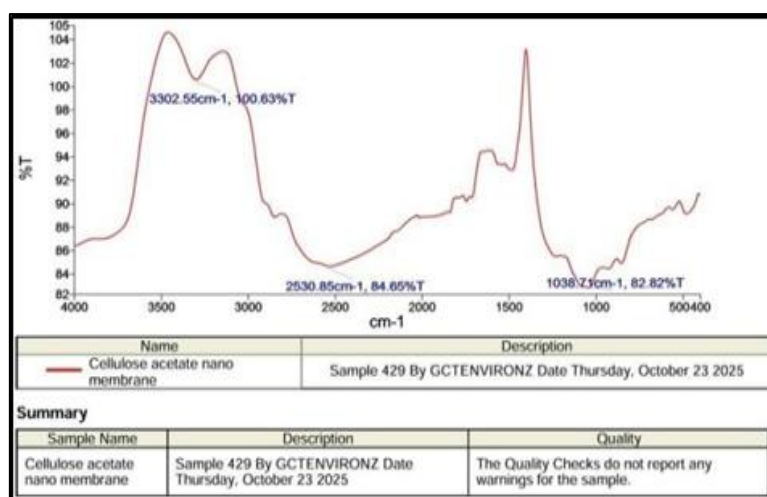
of the nanoparticles was calculated to be 225.4 nm which means the nanoparticles were successfully synthesized with average sizes within the nanometer scale. There is one dominant peak in the particle size distribution graph which represents a single particle population that can be observed with minimal clustering. The PDI value obtained was 0.284, which implies that the particle size distribution was narrow, thus suggesting a stable colloidal suspension and it can be effectively incorporated into the cellulose acetate matrix while preparing nanomembrane.



**Figure 2.** Particle Size Distribution of Green-Synthesized Fe<sub>2</sub>O<sub>3</sub> Nanoparticles Obtained from PSA Analysis

### 3.1.2 Fourier Transform Infrared Spectroscopy (FTIR)

Figure 3 illustrates the FTIR spectra of green synthesized Fe<sub>2</sub>O<sub>3</sub> nanoparticles. In this spectrum, the absorption peak at 580.98 cm<sup>-1</sup> represents Fe–O vibrations, which signifies that Fe<sub>2</sub>O<sub>3</sub> nanoparticles have been successfully synthesized. The broad band at 2518.59 cm<sup>-1</sup> corresponds to O–H vibrations and represents hydroxyl functional groups present in iron oxide nanoparticles due to the reduction of phytochemicals present in the pomegranate peel extract. The peaks present at 1810.86 cm<sup>-1</sup>, 1478.01 cm<sup>-1</sup>, and 1028.80 cm<sup>-1</sup> represent vibrations of C=O, C–O, and C–N functional groups, respectively. These functional groups occur in bioactive molecules in the plant extract and serve as natural reducers and stabilizers in the preparation of iron oxide nanoparticles. The presence of an additional peak for Fe–O vibrations at 481.32 cm<sup>-1</sup> also shows successful synthesis of Fe<sub>2</sub>O<sub>3</sub> nanoparticles.



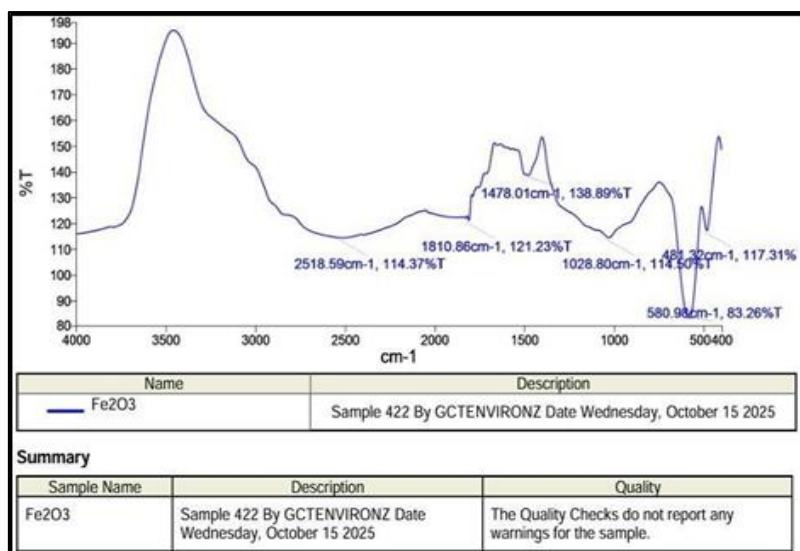
**Figure 3.** FTIR Spectrum of Green-Synthesized  $\text{Fe}_2\text{O}_3$  Nanoparticles Indicating Functional Groups Involved in Nanoparticle Formation and Stabilization

### 3.2 Characterization of Cellulose-Iron Nano Membrane

The obtained cellulose-iron nanomembrane was analyzed for its chemical structure, optical and thermal properties after incorporation of  $\text{Fe}_2\text{O}_3$  nanoparticles by various methods like FTIR, UV-Visible spectroscopy, and Thermogravimetric Analysis. From all these analyses it could be observed that a stable nanocomposite nanomembrane has been formed which is appropriate for the purpose of wastewater treatment using photocatalytic degradation.

#### 3.2.1 Fourier Transform Infrared Spectroscopy (FTIR)

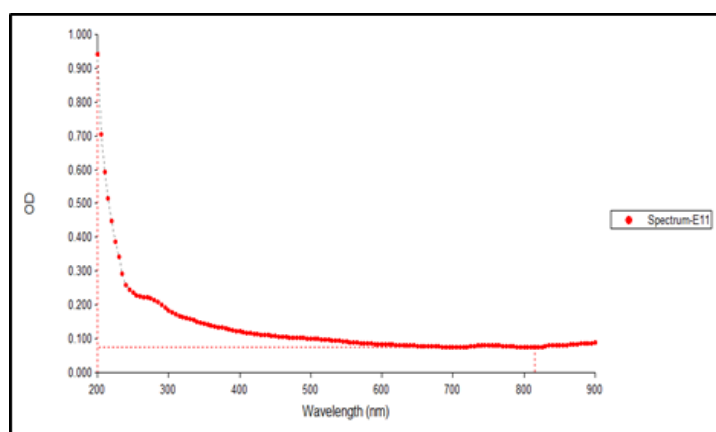
The FTIR spectrum analysis of cellulose acetate–iron nanomembrane clearly showed that  $\text{Fe}_2\text{O}_3$  nanoparticles were incorporated into the cellulose structure. In particular, there is a broad peak at 3302.55  $\text{cm}^{-1}$ , which is associated with O-H stretching vibrations in the membrane, showing the presence of hydroxyl groups in hydrogen bonds [25]. There are also peaks at 2530.85  $\text{cm}^{-1}$  (corresponding to C-H stretching) and a strong peak at 1038.71  $\text{cm}^{-1}$  (C-O-C stretching), which correspond to cellulose acetate [26]. The crucial point was that the change in the position and the intensity of these characteristic peaks, in comparison with cellulose acetate alone, was a clear sign of chemical interaction and binding between  $\text{Fe}_2\text{O}_3$  nanoparticles and polymer chain [25].



**Figure 4.** FTIR Spectrum of Cellulose–Iron Nanomembrane Confirming the Incorporation of  $\text{Fe}_2\text{O}_3$  Nanoparticles into the Cellulose Acetate Matrix

### 3.2.2 UV-Visible Spectroscopy (UV-VIS)

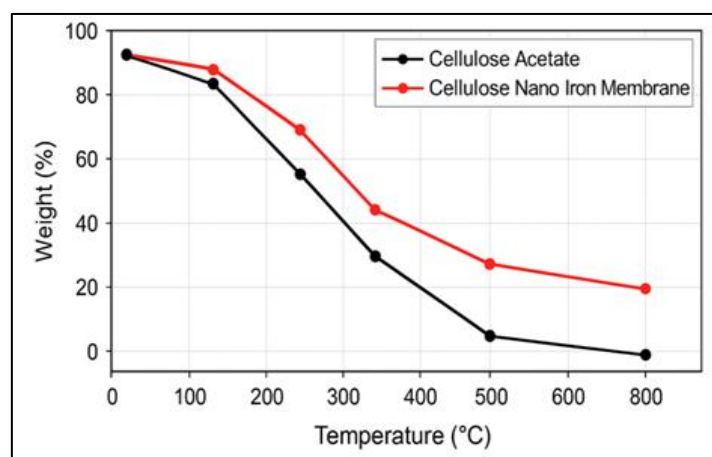
The UV-VIS absorption spectrum of the  $\text{Fe}_2\text{O}_3$  nanomembrane (Figure 5), displayed a strong absorption peak at 280 nm. The presence of the absorption peak is significant as it demonstrates the formation of iron nanoparticles in the cellulose matrix since it shows the surface plasmon resonance (SPR) of iron nanoparticles [27]. As demonstrated by other studies involving biosynthesis of  $\text{Fe}_2\text{O}_3$  nanoparticles, such results demonstrate an absorption range of 270 to 300 nm, thereby indicating the successful reduction of  $\text{Fe}^{3+}$  ions [28]. Besides, the strength of the absorption peak together with its shape suggests good dispersibility and high stability of the nanoparticles within the polymer matrix [27]. In conclusion, the UV-VIS absorption data clearly demonstrate the successful formation of iron nanoparticles in the form of homogeneous dispersion within the nanomembrane.



**Figure 5.** UV–Visible Absorption Spectrum of Cellulose–Iron Nanomembrane

### 3.2.3 Thermogravimetric Analysis (TGA)

The Thermogravimetric Analysis (TGA) curve Figure 6 clearly explains the impact of nanoparticle incorporation on thermal properties. Both the pure cellulose acetate and the cellulose–iron nanomembrane initially show a minor weight loss below 100°C due to moisture evaporation [32]. However, the pure cellulose acetate undergoes major degradation between 200°C and 400°C from the breakdown of its polymer chains, leaving only about 5% residue at 800°C.



**Figure 6.** Thermogravimetric Analysis (TGA) Curves of Pure Cellulose Acetate and Cellulose–Iron Nanomembrane

In sharp contrast, the cellulose-iron nanomembrane shows a remarkable increase in thermal stability, with its delayed weight loss and greater residual weight percentage (about 30%) observed even at 800°C [32]. Such an increased thermal stability and char yield is a clear result of the protective effect of iron nanoparticles that reduce the movement of the polymeric chains during decomposition.

## 4. Pharmaceutical Drug Removal Using Iron Nano Membrane–UV Photo Catalytic Reactor

The contamination of the environment by pharmaceutical chemicals has been found to be a major environmental problem in recent times. A large number of antibiotics like amoxicillin have been used in medicine and veterinary applications, and there is much leakage of these chemicals into aquatic environments from hospital effluents and industrial waste waters [20]. These chemicals are highly persistent and cannot be removed using the traditional method of water treatment. Water contamination by the residual chemicals leads to severe

problems like the development of antibiotic resistance bacteria, toxicity in aquatic organisms, and imbalance in the ecosystem [21]. In order to address this problem, novel and efficient methods of treatment need to be developed. In this regard, the use of iron nanomembranes in conjunction with UV reactor has been found to be an effective treatment technique.

#### 4.1 Principle of Operation

The iron nano-membrane/UV reactor technology works on the principle of an integrated system with adsorption, filtration through membranes, and UV-assisted advanced oxidation process [22]. Iron nanomembranes act as a first stage of treatment by removal of pollutants through adsorption and size-exclusion process, followed by UV exposure with wavelengths usually around 254 nm that help in degradation of any pharmaceutical residue in the wastewater. Iron nanoparticles either embedded in the membrane or dissolved in the solution assist in production of reactive oxygen species, mostly hydroxyl radicals ( $\cdot\text{OH}$ ), which are known for their ability to oxidize complicated organic matter [23].

#### 4.2 Mechanism of Pharmaceutical Drug Removal

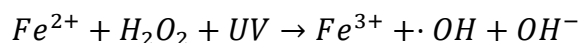
##### 4.2.1 Adsorption and Membrane Filtration

The iron nanomembrane is characterized by a high surface area owing to its nanosized structure. The pharmaceuticals get adsorbed to the surface of the membrane via the physical and chemical adsorption mechanism [32]. In addition, the nanomembrane itself works as a physical separator to remove any suspended particles.

##### 4.2.2 UV- Photocatalytic Degradation

When subjected to UV light exposure, the formation of active radicals is promoted by the iron nanoparticles. The reactive radicals then interact with the drugs and promote oxidative reactions that bring about their destruction [36].

The simplified reaction mechanism can be expressed as:



Where, hydroxyl radicals produced are highly reactive and capable of breaking down complex molecular structures present in pharmaceutical compounds.

### 4.2.3 Experimental Procedure

In the first step, a solution of amoxicillin pharmaceutical with a definite concentration was prepared [18]. The pH of the solution was then adjusted to a certain value to create the best conditions for the experiment. Then, the prepared solution was made to pass through the iron nano membrane in order to remove bulk materials and improve the effectiveness of the next stage [20]. The filtrate from the iron nano membrane was collected in the UV reactor where the solution was treated using ultraviolet rays with a wavelength of 254 nm continuously stirred [28]. This experiment was done for a period of 20, 40, and 60 minutes respectively in order to get samples. The samples were further filtered in order to eliminate any suspended materials. The absorbance of these solutions was finally determined using the UV–VIS spectrophotometer.



**Figure 7.** Experimental Setup of the Iron Nanomembrane–UV Photocatalytic Reactor Employed for Amoxicillin Degradation Studies

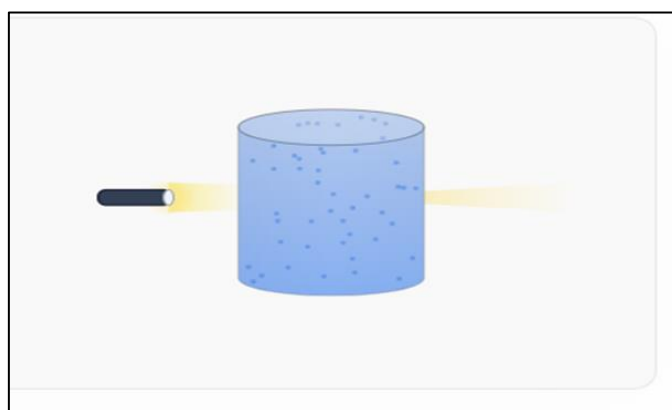
The setup of the cellulose-acetate membrane coated with iron nanomembranes for the study of photodegradation process of pharmaceuticals using UV radiation is shown in Figure 7. In the current study, an apparatus for the photocatalytic degradation of pharmaceuticals is employed that comprises a reactor with UV light source integrated together with fabricated cellulose-acetate iron nanomembranes for adsorption, filtration, and degradation processes.

$\text{Fe}_2\text{O}_3$  nanoparticles contained in cellulose acetate film under UV radiation become activated, forming electron-hole pairs. Interaction of these carriers with water and molecular oxygen leads to the formation of highly active reactive species, namely hydroxyl ( $\bullet\text{OH}$ ) and superoxide radicals ( $\text{O}_2^{\bullet-}$ ). The radicals formed initiate oxidation processes of amoxicillin molecules, which leads to their decomposition into simpler substances. Besides the

photocatalytic effect, the presence of a nanomembrane allows increasing adsorption of pollutants by the membrane structure.

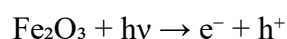
## 5. Result and Discussion

The experimental arrangement used to carry out the experiments on amoxicillin photodegradation is shown in Figure 8. It includes a UV lamp and a cell filled with the cellulose–iron nanomembrane. As a result of UV illumination, Fe<sub>2</sub>O<sub>3</sub> nanoparticles undergo photocatalysis that leads to the formation of reactive oxygen species degrading amoxicillin molecules.

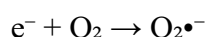
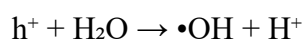


**Figure 8.** Degradation Efficiency of Amoxicillin Using the Cellulose–Iron Nanomembrane Under Different

The absorbed photon energy generates electron–hole pairs according to



The generated holes react with water molecules to produce hydroxyl radicals, while the electrons interact with dissolved oxygen to generate superoxide radicals, as represented by



The subsequent oxidation of amoxicillin by these very reactive species results in the transformation of amoxicillin into less complex intermediates until mineralization is achieved through conversion into carbon dioxide, water, and ions. The obtained value of 90% degradation efficiency after 60 minutes proves the high potential of using the cellulose-iron nanomembrane-UV photocatalytic reactor in treating pharmaceutical wastewaters.

The degradation efficiency of the pharmaceutical compound is calculated using the following equation:

$$\text{Degradation Efficiency (\%)} = \frac{C_0 - C}{C_0} \times 100$$

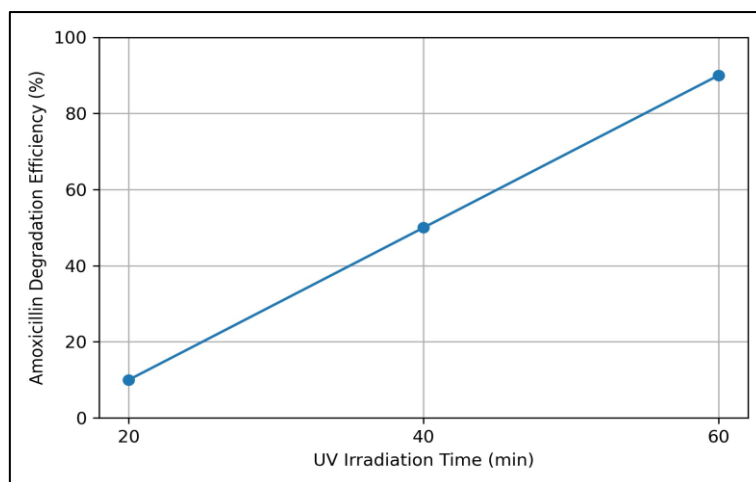
Where:

- $\eta$  = Degradation efficiency (%)
- $C_0$  = Initial amoxicillin concentration
- $C$  = Concentration at time

**Table 2.** Degradation Efficiency of Amoxicillin Under UV Irradiation

<b>Irradiation Time (min)</b>	<b>Degradation Efficiency (%)</b>	<b>Observation</b>
20	10%	Initial degradation observed due to adsorption of amoxicillin molecules onto the cellulose–iron nanomembrane surface and the onset of photocatalytic activity.
40	50%	Significant increase in degradation efficiency resulting from enhanced generation of reactive oxygen species and improved interaction between amoxicillin molecules and Fe <sub>2</sub> O <sub>3</sub> catalytic sites.
60	90%	Maximum degradation efficiency achieved due to prolonged UV exposure, leading to continuous hydroxyl radical generation and effective photocatalytic oxidation of amoxicillin.

The efficiency of degradation of amoxicillin in the presence of cellulose-iron nanomembrane under various UV exposure periods is shown in Table 2. It is evident that as the UV irradiation period increased, the efficiency of degradation also increased progressively. This improved degradation efficiency is due to the combined effect of membrane adsorption and photocatalytic oxidation.



**Figure 9.** Effect of UV Irradiation Time on the Degradation Efficiency of Amoxicillin Using the Cellulose–Iron Nanomembrane

According to Figure 9, the effectiveness of degradation started rising gradually starting from 10% at 20 min up to around 50% at 40 min. An additional extension of irradiation time up to 60 min led to a degradation efficiency approaching 90%, which proves high photocatalytic activity in the created membrane material. At first, degradation is explained by an adsorption process when amoxicillin molecules adhere to the membrane and generate a small amount of reactive species. With the rise in irradiation time, more electron-hole pairs emerge in  $\text{Fe}_2\text{O}_3$  nanoparticles and increase the formation of hydroxyl ( $\bullet\text{OH}$ ) and superoxide ( $\text{O}_2\bullet^-$ ) radicals, attacking the pollutants and promoting the oxidation reaction.

High photocatalytic activity in the system was obtained due to the combined effect of adsorption and photocatalytic processes. Cellulose acetate is characterized by its highly porous structure and large specific surface, which leads to high pollutant adsorption on the membrane. In addition,  $\text{Fe}_2\text{O}_3$  nanoparticles, which were introduced in the structure, possess photoactive properties under ultraviolet radiation. The combined effects of adsorption and photocatalysis provide efficient interaction of pollutants with catalytic centers and, therefore, higher degradation.

## 6. Conclusion

The current study successfully achieved the green synthesis of iron oxide nanoparticles from *Punica granatum* peel extract and their successful incorporation into the cellulose acetate nanomembrane via electrospinning process. Results obtained from characterization analysis confirmed the successful formation of iron oxide nanoparticles and their uniform dispersion in

the membrane matrix with improved physicochemical characteristics of the developed nanocomposite membrane. The cellulose iron nanomembrane proved highly efficient in photocatalytic activity when used in combination with the UV reactor; around 90% degradation of amoxicillin could be achieved in 60 minutes of irradiation. It is concluded that the use of membrane for the adsorption along with photocatalytic oxidation process considerably improved the effectiveness of removal of pharmaceutical contaminants from wastewaters. Utilization of agricultural waste products for the synthesis of nanoparticles increased the environmental friendliness of the proposed method. Further research efforts might concentrate on the investigation of membrane stability and its reusability in several photocatalytic reaction runs, degradation of various pharmaceutical contaminants, optimization of reaction parameters, and pilot-scale experiments involving actual wastewater.

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