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RESEARCH ARTICLE

Comprehensive Analysis of γ-Radiation Impact on PVA-PTh Composite Films for Enhanced Battery Functionality

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Abstract

Objectives: This research serves to explore the dynamic interaction between γ-radiation and PVA-PTh (Polyvinyl alcohol-Polythiophene) composite films with the intent to revolutionize the field of rechargeable batteries. The objective of this study is synthesis of PVA-PTh films and to comprehend the impacts of γ-radiation at various dosage rates with greater emphasis to their electrical characteristics. **Methods:** Our methodology encompasses the application of a chemical oxidative technique to form PVA-PTh nanocomposite films, followed by an enlightening analysis through SEM, FTIR, AAS, and electrical conductivity measurements after γ-radiation. The fascinating outcomes extend beyond the lab, with the PVA-PTh composite films and their γ-radiated counterparts venturing into the realm of battery development. **Findings:** The results unveiled are captivating. SEM analysis reveals the emergence of smaller, intricately structured flakes in γ-radiated PVA-PTh composite films, indicating significant morphological changes. FTIR results showcase shifts and new absorption bands at 788 cm^{-1} and 1031 cm^{-1}, evidence of chain crosslinking and scission processes triggered by γ-radiation. Surprisingly, although this radiation reduces the iron (Fe) ion content in PVA-PTh films, the electrical conductivities remain unwavering, even when exposed to 30 kGy (kilo Grays) dose. This steadfastness extends to consistent open circuit voltage (Voc) of 0.30 volts, which persists for astonishing duration of approximately 35 minutes in PVA-PTh conducting films. This exceptional stability paves the way for integration of films into cutting-edge polymer battery systems. **Novelty:** This research unlocks opportunities for innovative applications, invites further exploration across diverse fields by providing groundbreaking insights into the morphology, chemical composition, electrical behavior of PVA-PTh composite films. The exceptional stability of electrical conductivity in γ-radiated PTh samples, even under the harshest radiation conditions, offering testament to their reliability and resilience. These results point towards modern polymer battery systems with consistent Voc and enduring current flow, supporting the PVA-PTh composite films in battery applications.

Keywords: PTh; γ-radiation; Polythiophene; PVA-PTh; Battery

1 Introduction

In recent decades, production and smart applications of nanocomposites are especially fascinating [1]. Polymers are good hosts for metal nanoparticles (NPs) because they can act as reducing agents during the synthesis of NPs and prevent the agglomeration of NPs. In addition to these essential qualities, the polymer matrix also gives the systems solubility, processability, and thermal stability [2].

Numerous polymers, including polyvinyl alcohol (PVA) [3,4], polyacrylic acid (PAA) [5], polyethylene glycol (PEG) [6], polyaniline (PANI) [7], PTh [8] and many co-polymers [9], have been found to be beneficial for sustaining these nanomaterials [10–12]. Due to its distinctive qualities, such as excellent film formation, high hydrophilic characteristics, easy processability, non-corrosive nature, good mechanical properties, and good thermal stability, PVA is one of the excellent host matrix for embedding metal nanoparticles, which has a lot of isolated hydroxyl functional groups that can bind and complex with metal ions [13]. Among these nanocomposites, the films composed of PVA and PTh have shown favourable outcomes. These films were prepared by *in situ* chemical oxidative technique [11].

Radiation based polymer modification is an innovative technique that has garnered a lot of interest. The ionizing radiations produce free radicals uniformly in the medium they travel through due to the extremely high energy of their photons (0.66-1.25 MeV) or accelerated electrons (several KeV to 10 MeV). This enables the irradiation of monomers or polymers in any state at room temperature or below, if necessary. Ionizing radiation is an effective technique for creating nanomaterials due to their unique characteristics [14]. The γ-radiation stands out as powerful weapon among the different ionizing radiation types used for this purpose because of its distinct properties and broad range of applications. Researchers may develop materials with desired properties for a number of purposes by precisely controlling the dosage and exposure period of γ-radiation to favour different reactions [15]. The characteristics of the polymer can be customized to fit certain applications by altering the dosage and exposure period to favour different reactions due to the potential uses in integrated circuits, sensors [9], optical devices [16], rechargeable batteries [17], etc.

In the previous work the authors such as Oberhaus F.V. et al. discussed the information about the electropolymerization of high-quality PTh films and then developed a gentle method for electrode generation [18], Thanasamy D. et al. reported the synthesis of greater yield of 80% for PTh with a maximum electrical conductivity of 9 Scm^{-1} at room temperature [19], Jose F. et al. studied the simultaneous *in situ* synthesis of PTh and silver nanoparticles embedded in a Polymethylmethacrylate (PMMA) matrix to form a conducting polymer [20]. Cherkasinha N.I. et al. presented the effect of γ-sources on polyimide composites with WO_2, studied experimentally the influence of γ-radiation (doses upto 10 MGy) on the samples using ^{137}Cs (E = 0.662 MeV) and $^{6\circ}Co$ (E = 1.252 MeV) sources [21]. Chikaoui K. et al. investigated the effects of γ radiation on the polymer polyethylene terephthalate (PET) using a variety of techniques, including Fourier Transform Infrared Spectroscopy (FTIR), X-ray diffraction (XRD), and UV-Visible spectrophotometry [15]. Al-hada N.M. et al. discussed synthesis of conducting PANI composite film from 10 kGy to 30 kGy doses at ambient surroundings using the γ-radiation method [22].

However, from the recent literature survey, it is observed that the effect of γ radiation on PVA-PTh composite films remains relatively unexplored. Hence, there is a compelling interest in investigating the impact of γ-radiation on the properties of PVA-PTh composite films.

We offer an original research work within the theme of PTh nanocomposites materials. Briefly, this paper reported the preparation of PVA-PTh nanocomposite film samples via the chemical oxidative technique. Also, the aim of this research is to find the effect of γ-irradiation of about 30 kGy at room temperature, which is very high as compared to existing research, characterized with analytical techniques, including FTIR spectroscopy, Atomic Absorption Spectroscopy (AAS), Scanning Electron Microscopy (SEM), and electrical conductivity measurements. The result indicates that the electrical stability of PTh films were enhanced for longer duration by exposing the γ-irradiation dosage. Furthermore, these PVA-PTh films that had undergone γ-radiation were utilized in battery applications [10,23] and measure their open circuit voltage (V_{OC}).

2 Material and Methods

2.1 Materials

Pure PVA was obtained from Loba Chemie Pvt. Ltd, Mumbai, India. Thiophene (Th) (99% pure) was obtained from Lancaster, made in England. These were used as monomer for synthesis. Anhydrous Iron(III) chloride ($FeCl_3.6H_2O$) (96 % pure) was obtained from S.D. Fine chemicals Ltd., Boisar, India. It was used as a strong oxidizing agent during the polymerization. The purity of all these chemicals was of analytical grade and utilized as received, without further purification. All solutions were prepared using double distilled water.

2.2 Methods

2.2.1 Synthesis of PVA-PTh composite films

In the present research, we had done synthesis of PTh films. The first step in this process is to make a 4% w/v concentration of PVA solution, which is accomplished by dissolving PVA powder in double distilled water at 70-80°C to produce a clear solution. Then, this solution is rapidly agitated at room temperature for about 30 minutes while freshly distilled Th is added in a variety of volume/weight ratios with respect to the PVA. The solution is subsequently chilled to a temperature between 0 and 5 °C, and then $FeCl_3$ is used as the oxidizer to cause Th to polymerize. For better result 1:1 M ratio is kept constant. The schematic diagram showing synthesis of PVA-PTh composite films is illustrated in Figure 2.

A homogeneous solution with a light greenish yellow colour is the result of this. This solution was poured onto flat glass or polypropylene petri dishes to produce films. The amount of solution used determines the film thickness, which is normally 6 ml to achieve a thickness of 30–40 mm. After allowing the solvent to naturally evaporate for 24 hours at room temperature, PTh films that are uniformly bright greenish yellow were produced. The chemical reaction underlying the PTh preparation method is commonly illustrated in Figure 1.

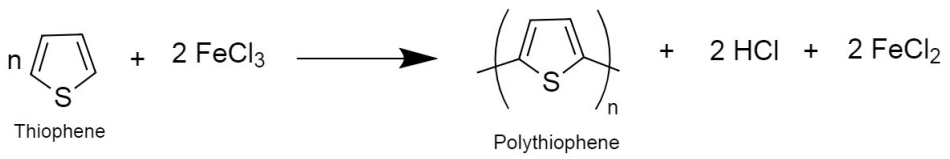

Fig 1. Chemical Reaction for the Synthesis of PTh

2.2.2 γ-irradiation

The films were sliced into 4 x 4 cm^2 sizes for γ-irradiation samples, mounted on a sample holder, and placed in the radiation zone of γ-radiation for characterization. The irradiation was performed at the Indian Institute of Technology's (IITs) Sophisticated Analytical Instrument Facility (SAIF) utilizing γ-chamber 5000. A ^{60}Co source was employed, which emitted γ-radiations with energies of 1.17 MeV and 1.37 MeV. Radiations from 1 to 80 kGy were used to irradiate the samples. Figure 2 illustrated a schematic describing the synthesis of γ-radiated PVA-PTh films.

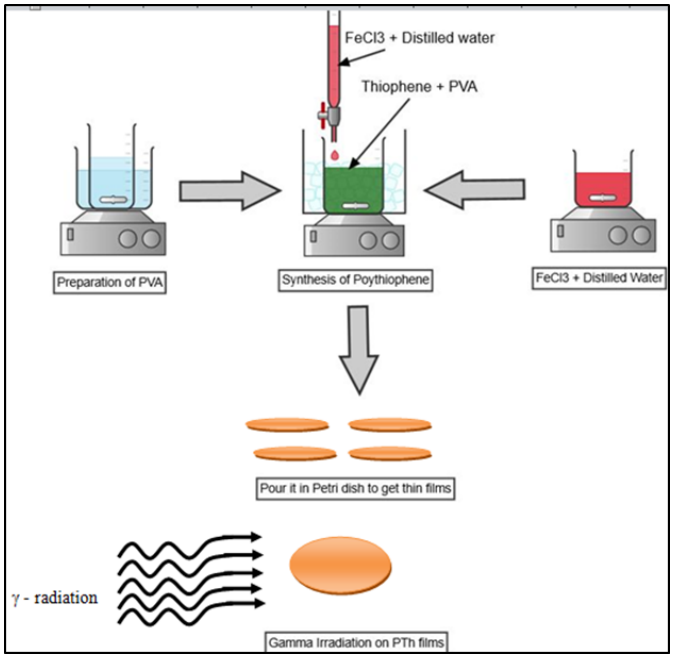

Fig 2. The schematic diagram for the synthesis of PVA-PTh composite films and exposure of γ-radiation on PVA-PTh films

2.2.3 Characterization methods and Electrical conductivity measurements:

Various analytical methods were employed to characterize the PVA-PTh composite and γ-radiation film samples. Surface morphological changes after irradiation were studied using the FEI Quanta 200 SEM. To analyze γ-irradiated samples, infrared (IR) spectra were analyzed using Attenuated Total Reflectance (ATR) on a FTIR spectrometer, specifically a Bruker ALPHA II FTIR Spectrometer, spanning the range of 4,000-400 cm^{-1} with 25 scans and a resolution of 4 cm^{-1}. The ARCOS Simultaneous ICP Spectrometer by SPECTRO Analytical Instruments GmbH, Germany, is used for AAS examination to determine Fe ion percentages. Finally, the electrical conductivity of the composite film samples is determined using the two probe method, with the formula,

Conductivity, $\sigma = \frac{V}{I}\frac{t}{A} = \frac{t}{R\,x\,A}$

Where, V = voltage applied, t = Thickness of the sample, I = current, R= resistance of the sample, A = Area of the sample.

3 Results and Discussion

3.1 SEM analysis

SEM examination of the globular morphology of several synthesized PVA-PTh samples is depicted in Figure 3. By employing a 4% aqueous solution to stainless steel plates, PVA films were produced. Figure 3 **a** makes it obvious that the control PVA film has an identical texture and smooth. Small globules or flake-like structures that visible on the surface of control PVA films suggest that these structures were aligned in a specific manner. The configuration of this structure can be seen visually and might provide information about the microscopic organization[24]. Such smooth film surfaces reduce the steric hindrance of eventually immobilized bioreceptors and as a result, could lead to enhanced target sensitivity[18].

Moreover, morphological modifications in the PVA-PTh films illustrated in Figure 3**b**, were observed when they were coated on stainless steel plates under both control and γ-irradiated environments. The control PVA film displays an average globule size of 1.19 μm, in addition to smaller globules, larger flake-like structures measuring about 10 to 15 μm in size were observed. In contrast, in the case of PTh composite films, a cauliflower-like structure was spotted over a significant area, along with fine particles embedded in the spherical structure. This implies that the morphology of the composite film is significantly influenced by the presence of PTh in it[19]. Each globule structure in the composite film has an average particle size of about 2 μm, which is less than the average globule size seen in the control PVA film.

The PVA-PTh films had significant morphological changes after being exposed to γ-rays, as seen in Figure 3**c**. Films exposed to γ-radiation showed the formation of flaky structures. These flaky formations were distinctive from the prior small globules perceived. These newly formed flaky structures were about 1 μm in size. This size is much smaller than the bigger flake-like structures found in the control PVA film (which ranged in size from 10 to 15 μm). The little globules that were previously present in the films persisted in addition to the newly created flaky structures.

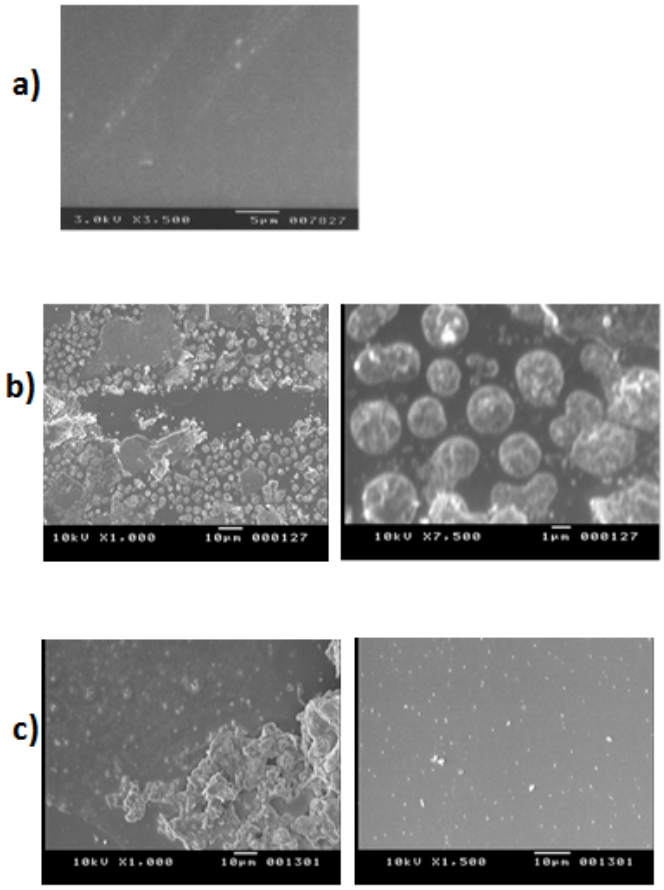

Fig 3. SEM images for a) Control PVA b) PVA-PTh films (Low and High magnification) c) PVA-PTh films irradiated with γ-rays

3.2 FTIR analysis:

FTIR spectra of two different materials—a composite film made of PVA mixed with PTh and its γ-ray exposed form can be found in Figure 4.

Within the spectrum region of 400 to 4000 cm^{-1}, the spectra were extensively examined, shedding light on the chemical composition and bonding characteristics of these materials. The Table 1 shows the peak positions associated with various major chemical bonds, as given in references [18,19,24].

At certain wavenumbers, distinct absorption peaks for the PVA-PTh film appeared, each one corresponding to important chemical bonds and molecular vibrations. Notably, a prominent peak in the 3330–3340 cm^{-1} region was observed, showing O–H stretching, a characteristic of hydroxyl groups. The peak at 2942 cm^{-1}, which reflected the alkyne bonds of C-H stretching vibration, was another distinguishing property [24], C-H out of plane stretching of α-α'coupling of PTh at 788 cm-1 [19]. There were other peaks at 1430 cm^{-1} (connected to CH_2 bending), 1141 cm^{-1} (related to C-C and C-O-C stretching), 1096 cm^{-1} (indicating C-O stretching), 916 cm^{-1} (highlighting CH_2 rocking), and 850 cm^{-1} (highlighting C-S bending) [19]. The C-H aromatic in plane bending vibration was also connected with weaker peaks seen at 1155 cm^{-1} and 1108 cm^{-1}, as well as an obscure peak at 1385 cm^{-1} that denoted the C=C stretch of the quinoid ring [24].

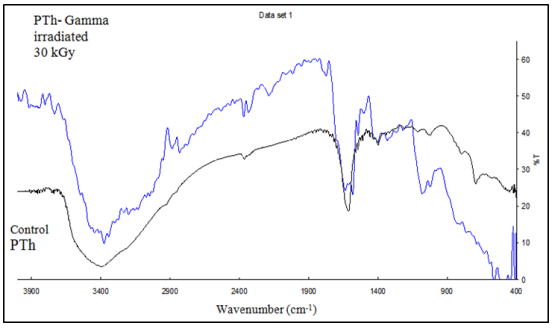

Fig 4. FTIR spectra of control PTh and γ-irradiated PVA-PTh film (30 kGy)

Table 1. Influence of γ-radiation on peak values of PVA-PTh films and comparison with past research

Vibrations (cm^{-1})	Past Research (cm^{-1})	PVA-PTh composite films (cm^{-1})	γ-radiated PVA-PTh films (30 kGy) (cm^{-1})
C=C stretching	1428 [19] 1649 [8]	1385	-
O-H stretching	3437 [8]	3336	-
C-O stretching	-	1096	-
C-C stretching	-	1141	-
C-S bending	731 [19]	850	788
C-H in plane bend	1155 [8,19]	1052	1031
C-H out plane bend	783 [19]	788	-

An intriguing trend became noticeable when these results were compared to the γ-irradiated film. The percentage transmission of the γ-irradiated film was significantly higher than that of the control PVA-PTh film. This FTIR spectrum deviation strongly suggests shifts or the formation of completely new absorption bands. Such spectrum alterations frequently denote chain crosslinking and scission processes that were probably triggered by a free radical mechanism imposed by γ-radiation.

3.3 AAS analysis

As per recent literature, rarely, the authors employed AAS to assess PTh composite samples. AAS is an effective analytical technique for determining the concentration of certain elements in a given sample. The Fe ion content of two sets of samples was investigated in our study, the control PVA-PTh film and the PVA-PTh film treated to γ-ray irradiation at a dose of 30 kGy. Our outcomes of analysis were displayed in tabular form in Table 2, highlighting the differences in the proportion of Fe ion content after radiation exposure. This study offers substantial insight into how radiation affects the Fe ion composition of these samples and is a novel application of AAS for PTh materials.

Table 2. AAS to find Percentage of Fe composition in the sample

Sample	Percentage of Fe
PVA-PTh (control) composite film	19.86%
PVA-PTh composite film- γ irradiation	16.34%

3.4 Current-Voltage (I-V Characteristics)

When the I-V characteristics of the control PTh and the PTh films treated to γ-radiation were analyzed at room temperature, it was found that there were only minimal changes in their electrical performance. We compare the electrical conductivities of the control PTh sample with the γ-radiated PTh sample in Table 3 in order to quantitatively evaluate these variations.

Incredibly, the γ-radiated PTh sample demonstrated exceptional stability in its electrical conductivity readings after being exposed to γ-radiation at a dose of 30 kGy. Although the electrical conductivity of γ-radiated sample is less than the control PVA-PTh, the γ-radiated sample shows excellent electrical stability even after an hour. Control PTh samples fails to show this

Table 3. Conductivity for control PTh and γ-irradiated films

Sample	Electrical conductivity (S/cm)
PTh control prepared	0.2316×10^{-1}
PTh prepared – 30 kGy	0.140×10^{-4}

stability after one hour due to the solubility of PVA in it. These findings highlight how resilient the material's electrical properties are even in the face of γ-radiation.

3.5 Use of PVA-PTh composite films in Battery application

In this context of research, the most significant experimental setup included 4% PVA-PTh composite films as vital components for the electrode. These composite films were essential to the study as they were carefully chosen for their distinctive characteristics and traits. To investigate electrochemical behavior, they were used in combination with an electrolyte solution made of iron chloride ($FeCl_3$). The primary objective was to explore this composite material's complex electrochemical behavior in relation to an electrochemical cell. The electrochemical cell was meticulously manufactured and configured to enable accurate regulation of the internal current flow. An essential component of the experimental setup that allowed for this control was the addition of an external resistance with a calibrated value of 100 ohms. In this regulated electrochemical environment, PVA-PTh composite film was the anode (positive electrode) and iron (Fe) was the cathode (negative electrode). From the Figure 6, it was revealed that a constant V_{OC} of 1.21 volts and a consistent current flow of 0.6 mA during the course of the 35-minutes observation period. Schematic representation of PVA-PTh film as a Battery application is also depicted in Figure 5.

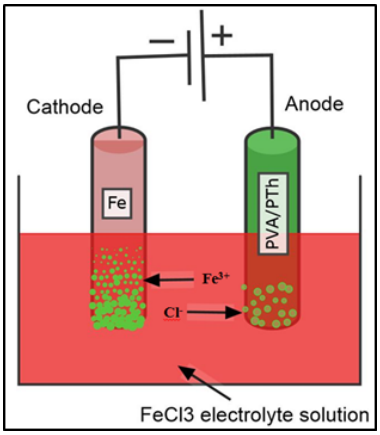

Fig 5. PVA-PTh film as an electrode showing the battery system

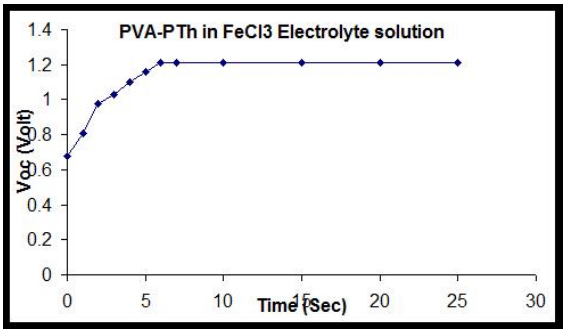

Fig 6. Response of Fe and PVA-PTh electrode in $FeCl_3$ electrolyte solution

The formula used to compute electrical power is,

$$P = I\,x\,V \quad (in\,Watt)$$

However, an immense barrier occurred when the PVA-PTh composite films were being assessed. These films showed a tendency for swelling after prolonged exposure to the aqueous media, which made them unusable as long-term electrodes in practical applications. Understanding how important it was to get overcome this obstacle, an intense effort was made to reduce PVA's solubility. In order to adjust the characteristics and solubility of the PVA-PTh composite films, γ-radiation was applied to them as part of the approach used to solve this problem. In particular, the composite films were exposed to a variety of dosages of γ-radiation, which ranged from 10 to 80 kGy, produced by a ^{60}Co radiation source. Although it was successful in slowing down the PVA-PTh composite films' rate of swelling, an unanticipated result was noticed. The electrochemical system output voltage, which had been 1.22 volts at first, declined significantly and was only 0.4 volts.

3.6 Use of PVA-PTh γ-radiated composite films in Battery application

In this study, we are primarily interested in investigating the potential that 4% PVA-PTh conducting films have as anodes for polymer battery systems. Despite its complexity, this research effort focuses especially on the characteristics of this performance of film after being exposed to γ-radiation, more precisely at a dose of 30 kGy. It does this by successfully combining cross-linking to decrease the solubility of PVA-PTh composite films with maintaining electrochemical behavior for battery applications. This 30 kGy optimal dosage selection offered consistent and reliable electrochemical performance within a radiation range of 10 to 80 kGy.

The experimental setup was carefully designed to evaluate the anodic properties of the PVA-PTh composite films. An electrolyte solution with $FeCl_3$ dissolved in distilled water was carefully used to aid in this evaluation. Additionally, in order to achieve accurate control over the current flow in our electrochemical setup, a 100 Ω calibrated external resistor was strategically incorporated. The average current flow in our experiment was found to be quite consistent at 0.576 mA and the V_{oc} stayed stable at 0.30 volts for an astonishing duration of around 35 minutes. The potential of 30 kGy radiation is highlighted by the accompanying graphical representation, which is presented in Figure 7 and graphically illustrates the battery response over time. Our research indicates that this radiation induced alteration optimizes the electrochemical performance of PVA-PTh conducting films, making them very suitable for modern polymer battery systems.

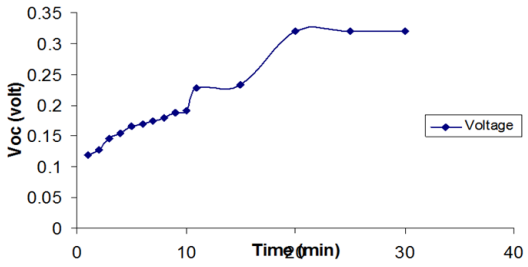

Fig 7. Response of Fe and γ-irradiated PVA-PTh electrode (30 kGy) in $FeCl_3$ electrolyte solution

4 Conclusion

The present study reflects a successful effort in the synthesis of PVA-PTh composite films and its exposure to γ-radiation, providing useful insights into their properties and potential applications. SEM analysis confirms different morphological changes in PVA-PTh composite films under both control and γ-irradiation conditions. The appearance of cauliflower-like structures as well as smaller flaky formations demonstrates specific influence of PTh on film morphology. The FTIR spectra of γ-irradiated PVA-PTh films reveal fascinating changes and the formation of new absorption bands due to probable chain crosslinking and scission processes, which are most likely initiated by free radicals generated by γ-radiation. To assess ion content in PTh composite samples, a novel application of AAS is presented. The study emphasizes the excellent electrical conductivity stability of γ-irradiated PTh samples, even after exposure to a high dosage of γ-radiation (30 kGy). The work focuses on the utilization of γ-radiated PVA-PTh composite films in battery applications and exhibits stable V_{oc} and current flow, establishing these films as an attractive option for contemporary polymer battery systems.

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