Honeycomb-like V$_2$O$_5$ Based Films: Synthesis, Structural, Thermal, and Optical Properties for Environmental Applications

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Abstract
In the present study, new composite films consisting of hierarchical nanobelt V$_2$O$_5$ and polymer mixture were prepared via a simple casting method. The incorporation of 30 wt.% of V$_2$O$_5$ into the polymer matrix yielded a honeycomb like structure with abundant micro-voids (5.5 μm), higher roughness average by 45.8%, and a higher root mean square roughness by 52%, which are beneficial for the enhancement of active surface area for dye adsorption. Furthermore, optical property studies have shown that the incorporation of V$_2$O$_5$ has made the nanocomposite film a suitable UV–visible light-sensitive material, and thus the application of films can be expanded towards photocatalytic degradation of various toxic pollutants such as nitrophenol, Cr(VI), antibiots, and so on. Finally, the composite film exhibited enhanced thermostability in comparison to unmodified film, as confirmed by TGA and DSC analysis. The optimal film showed 96.3% removal efficiency and 27.02 mg/g adsorption capacity. The dye sorption performance of V$_2$O$_5$ based films is studied at various times, dosages, and initial dye concentrations. The experimental data more closely fit the Langmuir isotherm model ($R^2 = 0.997$) than the Freundlich, Temkin, and Dubinin–Radushkevich isotherm models, demonstrating a monolayer adsorption mechanism. The MB adsorption process on V$_2$O$_5$ film was controlled by the chemical adsorption step, which was evidenced by the good-fitting of kinetic adsorption results to the pseudo second order model ($R^2 = 0.991$). The obtained results indicated that the V$_2$O$_5$ based films in this work are hopeful candidates for environmental applications.

Keywords V$_2$O$_5$ · Water treatment · Methylene blue dye · Adsorption process

1 Introduction
Pollution of water by pollutants such as dyes or pesticides has resulted in many of the environmental problems. In several places throughout the world, widespread industrial pollutants and wastewater containing organic dyes on land and aquatic bodies have resulted in serious contamination. During the dyeing process, around 1–20% of total global dye production is wasted and released into the environment as textile effluent. Toxic metabolites can be produced by weathering organic dyes through oxidation, hydrolysis, or other chemical reactions that occur in the wastewater phase [1, 2]. Furthermore, because available fresh water accounts for less than half of 1% of all the water on the planet, wastewater reuse and recycling are crucial. Every 20 years, global water use doubles, more than twice the rate of human population growth. Polluted water, particularly effluent from sludge, must be reclaimed to assist in ensuring that water resources do not run out [3, 4]. Methylene blue (MB) (3,7-bis(dimethylamino)-phenothiazin-5-i um chloride) is a thiazine cationic dye used in the dyeing of cotton, wood, cellulose, and silk. Despite its usefulness in science, the dye has negative consequences for humans and the environment due to its water solubility. It is poisonous, mutagenic, and thought to be a cancer-causing. Furthermore, MB may be hazardous to human health and lead to the development of chronic toxicity, particularly in the central nervous system. The textile industry’s effluent is heavily colored, contains high salt concentrations, and has high biological oxygen demand/chemical oxygen demand values. As a result, removing MB is critical for a better and healthier environment and health [5–8]. So, many researchers are working to discover effective techniques for eliminating...
these pollutants. Coagulation, chemical oxidation, adsorption, and photodegradation are some of the strategies utilized to treat wastewater [9–11].

Adsorption technique is one of the methods that widely employed in treatment of wastewater. Generally, adsorption is a surface phenomena in which a solution containing the adsorbate binds to the adsorbent's surface. Adsorption can take two forms; (1) physisorption, in which the adsorbate binds to the adsorbent due to van der Waals forces, and (2) chemisorption, in which the adsorbate adheres to the adsorbent due to chemical processes. Adsorption is thought to be a more efficient and cost-effective for dye removal approach than other wastewater treatment technologies. The fundamental benefit of this approach is that it produces high-quality effluent. Adsorption is a simple process that produces no hazardous pollutants, which makes it an environmentally beneficial method [12]. The difficulty of separating adsorbent material for reuse is a key disadvantage in the case of employing the adsorbent material in the powdered form for the wastewater treatment processes. As a result, using membrane/film technology to solve this problem is a viable option. This technology is thought to be the most effective way to alleviate water scarcity by supplying high-quality fresh water to a large number of people. This system has numerous benefits over traditional water treatment techniques, including cheap operating costs, reliability, high separation efficiency, simplicity, and environmental friendliness. As a result, waste treatment, the dyestuff industry, and food manufacturing have all used this technology [13]. In this aspect, the advancement of this technology relies heavily on the discovery of innovative materials. Over the last two decades, new research has concentrated on the development of synthetic membranes/films with enhanced characteristics for a variety of water treatment applications. Furthermore, problems to improve membrane long-term stability, durability, and cost efficiency have been identified. Various ideas using organic (polymers) or inorganic materials were developed for this purpose [14–16].

Vanadium is considered as one of the most ample transition metals in the earth’s crust. It has several valences from 2+ to 5+. developing different oxides such as VO, VO2, V2O5, and V2O3 [17]. Among these oxides, V2O5, known as vanadium pentoxide, is the most stable one through possessing the highest oxidation state (5+) and has a layered structure [18, 19]. To date, numerous researchers have developed V2O5-based compounds for diverse applications. Jayarat et al. [20] showed that V2O5 has antibacterial activity against Staphylococcus aureus and Escherichia coli bacteria. V2O5 [21], V2O5/MWCNT [22], and V2O5-nH2O-graphene oxide [23] were fabricated for lithium-ion batteries (LIBs) due to advantages of V2O5 including high theoretical capacity, simple synthesis, low cost, strong conductivity, good thermal stability, and extended cycle life [22, 24]. LIBs with high energy and good stability are becoming an essential part of our lives [25]. Wu et al. [26] synthesized NaV6O15 as a cathode material for the rechargeable magnesium batteries. V2O5/graphene [24] and WO3/V2O5 [27] composites were developed as an electrode material in supercapacitors. ZnO/V2O5 [28] and TiO2/V2O5 [29]. V2O5/PANI/GO [30] nanocomposites were synthesized for toluene, arsenic, and ammonia detection, respectively. Sreedhar et al. [31] reported the photoelectrochemical water splitting and NO2 sensing properties of V2O5/ZnO and V2O5/Co–ZnO films. Sribala et al. [32] modified polyimide with V2O5 for reduction of nitrophenol, Cr (VI), and rhodamine 6G dye. Dong et al. [33] sensitized N-doped TiO2 with various amounts of V2O5 for photodegradation of toluene. V2O5 was used for photocatalytic degradation of organic dyes such as Rhodamine 6G (Rh-6G), methylene blue (MB), and methyl orange (MO) [20] and Wang et al. [34] prepared TiO2/V2O5 for photodecomposition Rhodamine B (RhB) dye owing to that V2O5 is an n-type semiconductor with narrow bandgap (2.3–2.8 eV), non-toxic, unique surface characteristics, photo-stability, good chemical stability, excellent optical and light absorption properties [20, 33–35]. However such substantial efforts, developing V2O5 based films for removal of organic contaminants through either simple adsorption technique or photodegradation is seldom presented.

Herein, a series of composite films with various amounts of hydrothermally prepared V2O5 (0, 5, 10, 20, 30 wt.%) and a polymer making material were synthesized. Designing polymeric composite films based on V2O5 allows an easier recycling procedure. The introduction of V2O5 changed the surface morphology of pure polymer film from dense to a honeycomb like structure with enhanced thermostability. The reason for that could be the layer structure of V2O5, excellent filling action of V2O5, good compatibility, and homogenous distribution of V2O5 into the polymer matrix. The composite films acquire a dual functionality: (1) Enhanced dye adsorption properties, which are deliberated through removing MB dye as a model of wastewater contaminant, and (2) A visible light active material for photocatalytic processes, as revealed from optical properties studies. These characteristics prove their potential applications in wastewater treatment. The influence of different adsorption parameters on decolorization efficiency was deeply studied. Various isotherm models and adsorption kinetics were used to investigate the sorption mechanism.

2 Materials and Method

2.1 Materials

PEO (MW = 600,000) and PVDF (MW = 534,000) were purchased from ACROS and Merck, respectively.
Orthophosphoric acid, methylene blue (MB), and the dimethyl sulfoxide (DMSO) were provided from SD Fine Chemicals. Ammonium metavanadate (NH₄VO₃, 99%) was obtained from Sigma-Aldrich.

2.2 Preparation of Vanadium Oxide (V₂O₅)

V₂O₅ was prepared by the hydrothermal method as follows; the pH of 0.1 M NH₄VO₃ solution was adjusted to ~2.5 using orthophosphoric acid. The obtained solution was treated hydrothermally at 150 °C overnight. After cooling to room temperature, the precipitate was collected, washed with distilled water, dried at 70 °C overnight, and finally heat-treated at 500 °C.

2.3 Preparation of V₂O₅ based composite films

Different concentrations of the prepared V₂O₅ (0, 5, 10, 20, 30 wt.%) were incorporated into the polymer mixture solution, which consists of PEO/PVDF (25/75 w/w), at 65 °C using DMSO as a solvent. This solution was sonicated for 30 min and stirred for 6 h to enhance the dispersion of V₂O₅ particles into polymer matrix. After that the composite solution was poured onto Petri dishes and placed in a vacuum oven at 70 °C for around 12 h until the solvent had entirely evaporated. The samples are coded as x wt.% V₂O₅ where x is the concentration of V₂O₅ in the composite film relative to polymer content.

2.4 Measurement Techniques

X-ray diffraction (XRD) measurements were evaluated using Panalytical X’Pert PRO diffractometer with wavelength equals to 1.540 Å target Cu-Kα to confirm the crystal structure. JEM-2100F electron microscope with 200 kV accelerating voltage was used to perform a high-resolution transmission electron microscope (HRTEM) to show the shape of the prepared V₂O₅. The surface morphology and cross-section of the sample films were conducted using field-emission scanning electron microscope (FESEM) with EDX detector using Quanta 250 FEG. Pore size distribution of films was measured from FESEM images using ImageJ software. Using Gwyddion software (3D micrographs), the roughness characteristics were extracted from FESEM pictures. The Brunauer–Emmett–Teller ( BET) specific surface area was determined by using a Quantachrome apparatus, and the pore size distribution was estimated using the DFT method. Attenuated total reflection-fourier transform infrared (ATR-FTIR) analysis was performed on Bruker VERTEX 80, Germany. The absorbance of the produced films is measured in the range of 190–1000 nm using the V-770 UV–Visible/NIR spectrophotometer. The thermal stability of the prepared films was studied using differential scanning calorimetry (DSC, DSC131 evo, SETARAM Inc., France) from room temperature to 200 °C with heating rate 5 °C/min and thermogravimetric analysis (TGA, SDT Q600 V20.9 Build 20 thermal analyzer) with a heating rate of 10 °C/min from room temperature to 1000 °C.

2.5 Adsorption Experiments

Adsorption tests were performed in a batch mode reactor. For each experiment, 40 mg of adsorbent material was added to 40 ml of methylene blue dye solution 10 mg/L at room temperature (25 °C) under constant stirring (300 rpm). For monitoring the progress of the adsorption process, 3 ml of an aliquot solution was periodically taken out from the reactor and centrifuged at 6000 rpm for 2 min. At different times, the UV–visible absorption spectra of the collected filtrate were measured by using Shimadzu 2401 PC UV–Vis spectrophotometer, and the residual dye concentration was determined through the calibration curve previously made. All adsorption tests were carried out in duplicate and the average values are reported.

The adsorption percentage of the MB dye (% Adsorption) and the adsorption amount (qt) in the presence of various V₂O₅ based films are determined using the following equations.

\[
\text{% Adsorption} = \left(1 - \frac{C_t}{C_0}\right) \times 100\%
\]

\[
q_t = \frac{C_0 - C_t}{w} \times V
\]

where, C₀ and C₁ is the MB concentration at zero and specific time (t), respectively. V is volume of MB solution and w is the film weight. The impact of V₂O₅ content in the composite film on the dye decolorization efficiency was studied to determine the optimum film, which acquired the highest adsorption properties. After that in the presence of this sample, the influence of contact time ranged from 0 to 240 min and changing the initial dye concentration (10, 15, 20, 25, and 30 mg/L) were explored. The sorption data at equilibrium were fitted to the Freundlich, Langmuir, Temkin, and Dubinin-Radushkevich (D-R) isothermal models. The kinetics of the adsorption behavior of MB dye on films was studied using the pseudo first order, pseudo second order, intra-particle diffusion, and Elovich models to understand the mechanisms of the adsorption process.

2.6 Reusability

For water purification purposes, the utilization of films or membranes is characterized with the easy of recover after using. The reusability experiments were performed through

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immersion of the prepared films after the adsorption of MB in 0.1 M NaOH water:ethanol (40:60) solution overnight and washed with double distilled water for further use in adsorption–desorption process [36]. These regeneration experiments were conducted for four cycles.

3 Results and Discussion

3.1 Characterization of V₂O₅ and V₂O₅ Based Films

Figure 1 represents XRD patterns of the prepared V₂O₅ and various x wt.% V₂O₅ based films (0, 5, 10, 20, 30 wt.%). For powder V₂O₅, Fig. 1a, there are diffraction peaks at 2θ values 15.5°, 20.4°, 21.8°, 26.3°, 31.1°, 32.4°, 33.4°, 34.4°, and 41.4°, which are assigned to (200), (001), (101), (110), (301), (011), (111), (310), and (002) reflection planes. All peaks are consistent with JCPDS no. 01-070-8747 [37]. This result indicated that as-prepared V₂O₅ revealed orthorhombic structure with (001) predominant orientation, high purity, and good crystallinity. 0 wt.% V₂O₅ film has two diffraction peaks at about 18.8° and 19.7° as shown in Fig. 1b. For the V₂O₅ based films, Fig. 1c, d, e, f, we can see that there is a predominant bread peak appeared at ~20.6°. This peak may be attributed to the overlapping between the predominant (001) reflection of V₂O₅ at 20.4° and the peak associated to pure polymer film at ~19.7°. The intensity of this peak decreased and became broader with increasing the V₂O₅ content. No obvious diffraction peaks of V₂O₅ are detected in the XRD patterns after mixing with polymer mixture, which could be ascribed to the excellent compatibility between V₂O₅ and the polymer matrix as will be revealed from surface and cross section FESEM images. The incorporation of V₂O₅ in the composite film will be clarified by EDX analysis.

The morphology of pure V₂O₅ powder, which prepared by a template/surfactant free hydrothermal treatment using ammonium metavanadate and orthophosphoric acid, was examined by HRTEM as shown in Fig. 2a, b. It seen that V₂O₅ has hierarchical nanobelt interconnected structure with thickness in the range of 80–250 nm and 42–450 nm in length. Rong et al. [21] prepared V₂O₅ with comparable morphology through solid-state chemical reaction between ammonium metavanadate, tartaric acid, and polyethylene glycol 4000, and finally a calcination step. They found that V₂O₅ with hierarchical nanobelt structure as a cathode material for lithium ion batteries displayed low charge transfer resistance and excellent cycling stability. Also, Du et al. [23] reported that this architecture can enhance the interaction with organic materials and also avoid the aggregation of V₂O₅. Figure 2c shows the SAED pattern of V₂O₅, which verifies the presence of (200), (101), and (110) crystal planes in orthorhombic V₂O₅. These data confirm the XRD results of V₂O₅.

The morphological change owing to the incorporation of V₂O₅ into the polymer mixture was studied by examining the surface structure of the 0 wt.% and 30 wt.% V₂O₅ films by field emission scanning electron microscope (FESEM). Figure 3a displayed that 0 wt.% V₂O₅ film has a spherulite-like morphology [38] and its cross section image (Fig. 3c)
showed a smooth surface with no visible interface or phase separation between the two polymers, indicating good compatibility between PEO and PVDF [39]. Figure 3b shows that the film morphology is dramatically changed when V$_2$O$_5$ is mixed with the polymer matrix. It is known that V$_2$O$_5$ has a layered structure of VO$_5$ square pyramids that share edges and corners and are attracted by Van der Waals interactions [21]. From Fig. 3b, d, we can speculate that V$_2$O$_5$ particles are homogeneously distributed and interspersed among the chains of the polymer making film to form a honeycomb like structure as matched with the SEM images. This morphology may be attributed to interpenetrating architecture of V$_2$O$_5$, which enhances the interaction with organic materials and also avoids its aggregation [23]. The cross section image, Fig. 3d, proves the presence of abundant voids inside the V$_2$O$_5$ composite film. The micrograph, Fig. 3e, shows the mean diameter of the voids is about 5.5 μm. These higher internal pores usually provide a more active surface area, resulting in enhanced adsorption properties. Energy dispersive X-ray (EDX) was used to determine the elemental composition of the prepared 0 wt.% and 30 wt.% V$_2$O$_5$ films as shown in Fig. 3f, g, respectively. These spectra affirmed the presence of vanadium element within the composite film.

Figure 4 represents the 3D micrographs of 30 wt.% V$_2$O$_5$ film relative to the unmodified film (0 wt.% V$_2$O$_5$). The roughness of the surface is described by the roughness average (R$_a$) and root mean square roughness (R$_q$). These parameters have a significant impact on the physical adhesion of the produced films. Roughness heights operate as hooks, allowing films and their surroundings to interact more easily [40]. When compared with pure film, the incorporation of V$_2$O$_5$ increased the value of R$_a$ from 55.8 to 81.4 nm and also R$_q$ was increased from 71.1 to 108.1 nm. These increases are attributed to the development of microvoids after the addition of V$_2$O$_5$ to polymer mixture. This could increase the available active surface area, which consequently facilitate the adsorption of dye.

The size of the pores in the prepared membranes was also studied. Brunauer–Emmett–Teller (BET) specific surface area (S$_{BET}$) and pore size distribution of 30 wt.% V$_2$O$_5$ film in comparison with 0 wt.% V$_2$O$_5$ film were investigated.
by measuring the nitrogen adsorption–desorption isotherms as shown in Fig. 5. Figure 5a presented that both films display type-IV adsorption–desorption patterns. At P/P₀ > 0.5, the 30 wt.% V₂O₅ isotherm demonstrated a clear hysteresis loop of H₃ type, which assigned to capillary condensation and suggesting the development of mesoporosity [41, 42]. The specific surface area, respectively, was 42.23 m²/g and 63.25 m²/g for 0 wt.% V₂O₅ and 30 wt.% V₂O₅. As illustrated in Fig. 5b, the unmodified polymer film (0 wt.% V₂O₅) contains two sets of pores; the first one has a diameter ranging from 5 to 10 nm and there are minor quantities of pores with widths ranging from 20 to 30 nm. Whereas, the modified film (30 wt.% V₂O₅) has a greater number of pores. According to the pore size, these pores are divided into three
groups; 5 – 10 nm, 10 – 20, and 20 – 40 nm. Additionally, the incorporation of V2O5 causes a significant increase in the total pore volume from 0.0773 to 0.19000 cc/g. These results indicated that the V2O5 sensitization led to a significant increase in the textural parameters, including surface area, pore volume, and pore size, which might increase the adsorption capacity of organic dye by providing more active adsorption sites in the as-obtained 30 wt.% V2O5 film relative to pristine polymer film [42].

ATR-FTIR spectra were examined for the samples, including V2O5 and various V2O5 based films (0, 5, 10, 20, 30 wt.%) as shown in Fig. 6. Powder V2O5, Fig. 6a, has distinctive bands below 1010 cm−1. The bands appeared at lower than 830 cm−1 originating from V–O–V bending vibration and V–O–V edge-sharing vibration. The band at 1009 cm−1 is assigned to the V═O stretching vibration in V2O5 [35, 43–45]. This data indicates the successful synthesis of V2O5. ATR-FTIR spectrum of 0 wt.% V2O5 film, Fig. 6b, reveals the band at 2884 cm−1 due to the CH2 stretching vibration of PEO. The scissoring vibration of CH2 is seen at 1467 cm−1. The wagging vibrations of CH2 are seen at 1402 cm−1 and 1342 cm−1. The stretching vibration of C-F and CF2 are seen at 1179 cm−1. The stretching vibrations of C-O band are observed at 1104 cm−1 and 962 cm−1. Bands at 871 cm−1, 840 cm−1, 794 cm−1, 761 cm−1 and 533 cm−1 are assigned to the rocking vibration of CH2. The bending vibration of C-F is seen at 614 cm−1 [46–51]. These bands are attributed to the characteristic features of PVDF and PEO polymers forming the 0 wt.% V2O5 film. The ATR-FTIR spectra of various V2O5 based films are depicted in Fig. 6c, d, and e. As seen the bands at 2884 cm−1, 1342 cm−1, 1104 cm−1, and the C-O stretching vibration band at 962 cm−1 decreased in intensity with increasing the V2O5 content in the film. The bands at 794 cm−1, and 761 cm−1 are disappeared in the spectra of all composite films. There is a noticeable decrease in the sharpness of the band at 614 cm−1 after the incorporation of V2O5, and the band at 533 cm−1 is red-shifted to 508 cm−1. Finally, a new band appears at 990 cm−1, which is assigned to the V═O in V2O5 [35]. These results confirm the excellent interaction taking place between the polymer matrix and vanadyl groups of V2O5.
To assess the applicability of the newly synthesized composite films to act as photocatalysts, their optical properties were studied through the UV–visible measurements in the wavelength range of 190–1000 nm as shown in Fig. 7. The absorption spectrum of powder V$_2$O$_5$ is depicted in Fig. 7a. It exhibits absorption characteristics in both the UV and visible region, which is generally attributed to the electronic transition from O(2p) orbitals to V(3d) orbitals in V$_2$O$_5$ [44]. There are three absorption bands at 230–270 nm, 270–414 nm, and 414–580 nm as a result of three types of co-ordinations between V$^{5+}$ ions and oxygen in the V$_2$O$_5$ lattice, including tetrahedral, square pyramidal, and octahedral, respectively [20]. Whereas, the spectrum of 0 wt.% film shows only one weak band at about 200 nm originating from \(\pi \rightarrow \pi^*\) electronic transition in PVDF and PEO [52] and it doesn’t have any absorption properties in the visible region as presented in Fig. 7b. For the composite films, Fig. 7c, d, e, f the V$_2$O$_5$ bands in the wavelength range 200–550 nm are appeared. Furthermore, with increasing the V$_2$O$_5$ concentration, the cutoff wavelength is red shifted. This means that the photoactivity of the samples will be significantly enhanced. That too, the 30 wt. V$_2$O$_5$ film has extended absorption up to 1000 nm.

V$_2$O$_5$ is a direct band gap n-typ semiconductor [35]. Thus the optical energy gap of the samples was estimated by drawing \((\alpha h\nu)^2\) against the photon energy \(h\nu\) and then extrapolating the linear part of the plot to \(h\nu = 0\), Fig. 8. As presented in the inset of Fig. 8, the prepared V$_2$O$_5$ has an energy gap of 2.33 eV. This value corresponds well with other work [20]. It was observed that the optical energy gap of the films is decreased in the following order: 5.04, 3.41, 3.28, 3.12, and 2.88 eV with increasing the V$_2$O$_5$ content from 0, 5, 10, 20, and 30 wt.%, respectively. Hence, the synthesized V$_2$O$_5$ based films are suitable candidates for visible-light driven photocatalytic processes. Recently, photocatalysis is considered as a green technique through utilization of the abundant solar energy to get rid of the environmental pollutants through photocatalytic degradation of harmful and toxic organic contaminants and antibiotics from waste water. This process has numerous advantages such as fast, nondestructive, complete mineralization, inexpensive, and low temperature [20, 35]. There are various works presented
the photocatalytic activity of V2O5 based composites as a powdered material [20, 32–35], however as a film or membrane rarely to be found yet.

Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) techniques were used to study the thermal properties of 0 wt.% V2O5 and 30 wt.% V2O5 films. Thermal stability is an important feature of studying the behavior of polymer nanocomposites from the standpoint of their applications. The addition of nanofillers can greatly improve the thermal stability of the polymer matrix. Figure 9A shows TGA graphs of the samples, it can be seen that 0 wt.% V2O5 film loses 83% of its weight with increasing temperature up to 800 °C. In comparison, a 30% V2O5 film loses only 65.5% of its weight. These results suggested that the composite film exhibited enhanced thermal stability, which might be assigned to excellent thermal stability of the incorporated V2O5 [53]. 0 wt.% V2O5 film has three decomposition steps; the first decomposition ranged from 40–300 °C and was related to the small molecule splitting or volatilization, as well as moisture evaporation. The second decomposition step ranged from 300–417 °C which was related to PEO decomposition temperature and the third decomposition step ranged from 417–800 °C, which included the PVDF decomposition temperature. However, the V2O5 composite film has only two decomposition regions; the first one at 35–392 °C may be related to the elimination of residual water from the vanadyl group [54] and the second one ranged from 417–800 °C which is related the breakdown of polymer nanocomposites chains. Figure 9B presents the derivative thermogravimetric (DTG) graphs of 0 wt.% V2O5 and 30 wt.% V2O5 films. It is noticed that there are two decomposition temperatures at 377 °C and 460 °C in case of no filler, while 30 wt. % V2O5 film presents only one decomposition temperature at 490 °C. In overall comparison, V2O5 composite film showed higher degradation temperature and percentage weight residue relative to unmodified film.

DSC is a useful tool for determining the host polymer’s subsequent temperature transitions and relative changes in crystallinity. Figure 10 shows DSC thermograms of the prepared films. For pristine polymer film, 0 wt.% V2O5, there are two major endothermic peaks seen at 58 °C assigned to the melting temperature of PEO and at 158.1 °C which is related to the PVDF melting temperature [55, 56]. On the other hand, the 30 wt.% V2O5 film revealed only one melting temperature at about 162 °C confirming the miscibility between the polymer mixture and V2O5. The shift in melting temperature and...
increase in its intensity with the addition of V₂O₅ contributed to the improvement of thermal stability, implying that intermolecular interaction between V₂O₅ and the polymer blend was formed. The results of XRD and ATR-FTIR analysis confirmed the occurrence of such interactions. The addition of V₂O₅ to the polymer matrix also affects the crystallinity (χc) of the film, which is calculated as follows [57].

\[
χ_c = \frac{ΔH_{f}}{ΔH_{100}} \times 100
\]  

where ΔH_f is the enthalpy of the film and ΔH₁₀₀ denotes the enthalpy value of 104.6 J/g for the perfect PVDF [56]. The degree of crystallinity and thermostability of the composite film relative to the unmodified film may be due to the filling action and high thermal stability of the added filler (V₂O₅).

3.2 Dye Decolorization Studies

The main purpose of our work is to extend the application of the new synthesized V₂O₅ composite films towards wastewater treatment, particularly for removal of organic matters such as methylene blue (MB) dye through adsorption mechanism. The V₂O₅ content in the composite film, contact time, film dose, and dye concentration were taken into account to achieve the highest decolorization efficiency, as will be presented.

3.2.1 Effect of V₂O₅ Content in the Composite Film

Preliminary tests for studying the adsorption properties of the prepared V₂O₅ based films in comparison with the unmodified film were carried out using methylene blue (MB) as a model dye. The UV–Vis spectra of dye solution at specific interval times (0, 30, 60, 90, and 120 min) in the presence of various samples are shown in Fig. 11a, b, c, d, e. These spectra clearly indicate that the characteristic absorption band of MB at 665 nm decreases progressively after the incorporation of V₂O₅ in the polymeric film. Therefore, the elimination of the colourant material from water solutions is enhanced. The qualitative adsorption percentage of the MB dye was calculated using Eq. 1 and the results are presented in Fig. 11f. It was found that with increasing the V₂O₅ content in the film from 5, 10, 20 and 30 wt.% the adsorption properties were enhanced and the adsorption percentage was 59.8, 67.7, 90.3 and 96.3%, respectively. It is clear that the film contains 30 wt.% V₂O₅ showed the most effective adsorption of the MB dye relative to other samples.

In conclusion, to achieve high dye removal efficiency, the film with V₂O₅ of 30 wt.% was chosen to be used in the subsequent tests.

3.2.2 Effect of Sorption Time

Variation of adsorption time to reach equilibrium state was studied using the most active composite film, which containing 30 wt.% V₂O₅. The enhancement of dye removal percentage as a response to the adsorption time increase is revealed in Fig. 12. For experiment with C₀ = 10 mg/L and 40 mg of sample, the decolorization reached 96.3% after 120 min, whereas at higher sorption time, there is a slight increase in the dye removal up to 97.1%. These results indicate that 120 min is the equilibrium time for 30 wt. % V₂O₅ film under these adsorption conditions. Previous reports [58, 59] presented that after equilibrium time no noticeable adsorption takes place.

3.2.3 Effect of Film Dosage

Figure 13 shows the influence of using different dosages, including 20, 30, 40, and 60 mg of the optimum sample (30 wt.% V₂O₅ film) on the MB sorption efficiency. To achieve a cost-optimal dye adsorption process, the minimum absorbent amount that gets efficient dye removal must be taken into consideration. Generally, the dye adsorption efficiency improved as the film dosage increased as presented in Fig. 13. At lower doses of 20 and 30 mg the dye removal was 42.4 and 75.4%, respectively. However, when using 40 and 60 mg doses, it was significantly increased to 96.3 and 98.4%, respectively. This is owing to increasing the valid adsorption sites at high dosages, which consequently leads to a higher removal percentage. These data conclude that 40 mg is the optimal amount for efficient MB adsorption, which was used in the next experiments. Other studies [60, 61] concluded that there was no significant change in the dye removal efficiency after the optimal dosage,
Fig. 11  The UV–vis spectra of MB solution using the prepared films a 0 wt.%, b 5 wt.%, c 10 wt.%, d 20 wt.%, and e 30 wt.%. f The effect of V$_2$O$_5$ content in the film on the MB adsorption percentage. Adsorbent: 40 mg, volume: 40 ml, initial MB concentration: 10 mg/L.
which was attributed to the saturation of the available active sites on the surface of the adsorbent material.

### 3.2.4 Effect of Initial MB Concentration

In Fig. 14, the percentage of dye elimination over initial concentration (5, 10, 15, 20, 25, and 30 mg/L) was investigated in the presence of 30 wt.% V₂O₅ film. It is observed that increasing initial dye concentration led to a clear decrease in the dye removal efficiency owing to limited adsorption sites in the film. The sorption positions are sufficient for complete removal of MB at low dye concentrations, as shown by C₀ = 5, 10, and 15 mg/L. In other words, with increasing the dye concentration to 30 mg/L, the adsorption sites in the 30 wt.% V₂O₅ film couldn’t capture all the MB molecules, resulting in the lower percentage of adsorption (83.9%). These outcomes agree with the literature [62, 63].

### 3.3 Film Regeneration

Development of recyclable materials for efficient treatment of polluted water is a challenging task. Generally, membranes or films are characterized by their regeneration ability, avoiding the cost and time consumed in the case of using common adsorption processes; consequently it has an excessive influence on the development of large scale wastewater treatment [62]. Herein, the regeneration performance of 30 wt.% V₂O₅ film was studied as follows: after using the film for MB adsorption, it was removed from dye solution, immersed in NaOH solution, washed with double distilled water, and dried to be reused again. Figure 15 shows that the 30 wt.% V₂O₅ film achieved 96.3% removal of MB dye in the first run. It is clear that after four adsorption-regeneration cycles, the film still possessed good adsorption properties (85.3%).

### 3.4 Adsorption Isotherms

Freundlich, Langmuir, Temkin, and Dubinin–Radushkevich isotherm models were utilized to describe the MB sorption at equilibrium and its equations are summarized in Table 1.

Where qₑ and Cₑ are adsorption amount and dye concentration at equilibrium, respectively. C₀ is the initial concentration of MB dye and n refers to the heterogeneity factor. Kᵢ and Kₑ are the Freundlich constant and the Langmuir isotherm constant, respectively. q_max is the maximum
adsorption quantity estimated from the model. $R_L$ is the separation factor and is calculated using Eq. 6. $R$ is the universal gas constant, $T$ is the temperature, $A$ resembles to equilibrium binding constant, and $b_T$ is the Temkin isotherm constant (J/mol). $K$ is related to the mean adsorption free energy ($mol^2/kJ^2$), and $\epsilon$ (kJ$^2$/mol$^2$) is the Polanyi sorption potential, which calculated using Eq. 9. $E_{ads}$ is the adsorption free energy (kJ/mol) and is calculated using Eq. 10 [58, 66–68].

Figure 16 shows the Freundlich isotherm of 30 wt.% V$_2$O$_5$ film. From the intercept of the straight line, the value of the Freundlich constant ($K_F, mg/g) (L/mg)^{1/n}$ was determined to be 15.7. Additionally, the slope is equal to $1/n = 0.39$ indicating that the MB adsorption on the 30 wt.% V$_2$O$_5$ film is favorable. Adsorption is considered favorable at $1/n < 1$ and unfavorable at $1/n > 1$ [58]. The correlation coefficient ($R^2$) value was 0.904, demonstrating that the Freundlich isotherm model isn’t a suitable model to describe the adsorption data at equilibrium.

Figure 17 illustrates the Langmuir adsorption isotherm of 30 wt.% V$_2$O$_5$ film. The Langmuir constant ($K_L, L/mg$) was determined to be 2.3. According to the high value of correlation coefficients ($R^2 = 0.997$), the Langmuir isotherm model is the best to describe the adsorption of MB dye on V$_2$O$_5$ based films. Additionally, the value of the theoretical maximum adsorption capacity (mg/g) was 26.9, which was determined from the slope of the straight line. This value is close to the experimental adsorption quantity ($q = 25.1$ mg/g). Finally, the value of the separation factor ($R_L = 0.014$), which was calculated using Eq. 6, indicated that the adsorption of MB on 30% V$_2$O$_5$ film is desirable [66].

The Temkin isotherm assumes that the interaction between adsorbent molecules and adsorbate leads to a decrease in the heat of adsorption [58, 69], which is shown in Fig. 18. The Temkin isotherm constant ($b_T$) is equal to 483.5 J/mol and the equilibrium binding constant (A) is

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**Table 1** The equations of the Freundlich, Langmuir, Temkin, and Dubinin–Radushkevich isotherm models

| Isotherm Type       | Equation                                                                 |
|---------------------|---------------------------------------------------------------------------|
| Freundlich isotherm | $\ln q_e = \ln K_F + \frac{1}{n} \ln C_e$                                  |
| Langmuir isotherm   | $\frac{C_e}{q_e} = \frac{1}{q_{max} K_L} + \frac{C_e}{q_{max}}$             |
| Temkin isotherm     | $q_e = \frac{RT}{b_T} \ln A + \frac{RT}{b_T} \ln C_e$                     |
| Dubinin–Radushkevich isotherm | $\ln q_e = \ln q_{max} - K e^2$                                      |
|                     | $n = RT \ln(1 + \frac{1}{C_e})$                                           |
|                     | $E_{ads} = \frac{1}{\sqrt{2K}}$                                            |

---

**Fig. 15** Adsorption percentage of MB dye in the presence of 30 wt.% V$_2$O$_5$ film for 4 cycles. Adsorbent: 40 mg, volume: 40 ml, initial MB concentration: 10 mg/L, and time: 120 min

**Fig. 16** Freundlich isotherm plot for adsorption of MB dye over 30 wt.% V$_2$O$_5$ film

**Fig. 17** Langmuir isotherm plot for adsorption of MB dye over 30 wt.% V$_2$O$_5$ film
equal to 30.26 L/g, which were determined from the slope and intercept, respectively. The value of the correlation coefficient ($R^2 = 0.983$) shows that this model isn’t well fitted to the adsorption data.

Dubinin–Radushkevich (D–R) isotherm model is shown in Fig. 19. $K$ is related to the mean adsorption free energy and equals to $4.3 \times 10^{-8}$ mol$^2$/kJ$^2$. The calculated maximum quantity adsorbed using this model is 22.19 mg/g. The free energy of adsorption ($E_{ads}$), which is calculated using Eq. 10, equals 3.39 kJ/mol. This value indicates that the adsorption process can be described as a physical process. The value of $R^2$ (0.977) indicates that the Dubinin–Radushkevich isotherm didn’t well fit the adsorption of MB on the 30 wt.% $V_2O_5$ film.

### 3.5 Adsorption Kinetic Studies

Under non-equilibrium conditions, the adsorption results of MB dye on 30 wt.% $V_2O_5$ film were fitted to three kinetic models; including pseudo-first order (PFO, Eq. 11), pseudo-second order (PSO, Eq. 12), and intraparticle diffusion (Eq. 13) [58, 70–73]. The fitted curves of these models are depicted in Fig. 20.

**Pseudo-first order:**

$$\ln \left( q_e - q_t \right) = \ln q_e - K_1 t \quad (11)$$

**Pseudo-second order:**

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e} \quad (12)$$

**Intraparticle diffusion:**

$$q_t = K_i t^{1/2} + C \quad (13)$$

where $q_e$ (mg/gm) is the adsorption amount at equilibrium, and $q_t$ is the adsorption amount at time $t$. $K_1$, $K_2$, $K_i$ are the PFO rate constant (min$^{-1}$), the PSO adsorption rate constant (g/mg.min), and the intraparticle diffusion rate constant (mg/g.min$^{0.5}$), respectively. The value of the correlation coefficient ($R^2$) was determined to compare the validity of each model. From Fig. 20, it is clear that the second model is the most fitted model with the largest $R^2$ value (0.991). This suggests that the MB adsorption by 30 wt.% $V_2O_5$ film is controlled by a chemical adsorption step [58, 73]. Moreover, the calculated amount adsorbed using this kinetic model (27.02 mg/g) is very close to the experimental one (25.1 mg/g).

### 4 Conclusion

To sum up, orthorhombic $V_2O_5$ with a hierarchical nanobelt structure was prepared via a surfactant free hydrothermal method. After that, for the first time, we designed $V_2O_5$ based films to huddle up the drawbacks of employing the adsorbent material in the powdered form for wastewater treatment. The composite films were prepared by a simple solution casting process through the incorporation of various amounts of the hydrothermally prepared $V_2O_5$ into a polymer solution. The adsorption properties of the $V_2O_5$ based films were evaluated for methylene blue (MB) dye removal. The prepared samples are characterized by XRD, ATR-FTIR, HRTEM, FESEM, EDX, BET, UV–Vis, DSC, and TGA analysis. The film’s cross section FESEM image demonstrates that $V_2O_5$ particles are homogeneously integrated with the polymer matrix to form a honeycomb-like
structure. The surface area, pore size, and total pore volume were all increased after the inclusion of V\textsubscript{2}O\textsubscript{5}. Additionally, incorporation of V\textsubscript{2}O\textsubscript{5} made the film act as a visible light sensitive material, enhanced the crystallinity, and improved the thermal stability. The experimental data are more fitted to the Langmuir isotherm model (R\textsuperscript{2} = 0.997) than Freundlich, Temkin, and Dubinin–Radushkevich isotherm models, demonstrating a monolayer adsorption mechanism. The MB adsorption process on V\textsubscript{2}O\textsubscript{5} film was controlled by the chemical adsorption step, evidenced by the good-fitting of kinetic adsorption results to the pseudo second order model (R\textsuperscript{2} = 0.991). The reusability tests indicated that the V\textsubscript{2}O\textsubscript{5} film was capable of removing up to 85.3% after four cycles.

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**Declarations**

**Conflict of interest** The authors declare that they have no competing interests.

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