Nanocomposites For Optical Properties and Effective Photocatalytic Degradation of Pollutants in Water

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Nanocomposites for optical Properties and Effective Photocatalytic degradation of pollutants in water

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Abstract

In this study, chromium (III) oxide (Cr_2O_3) and potassium oxide (K_2O) nanoparticles (NPs) were synthesized by combustion. Mixed oxides of varying concentrations in the form of $(1-x)\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ ($x = 0.1, 0.3, 0.5$, and 0.7) were prepared. X-ray diffraction (XRD), high-resolution transmission electron microscopy (HR-TEM), and optical measurements were used to investigate the properties of these nanoparticles. XRD indicates that the grain size decreased as the K_2O content increased. Additionally, HR-TEM revealed several types of shapes, such as hexagonally-shaped nanoparticles. The optical measurements showed a blue shift indicating a minor modification to the band structure during the $\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ mixing. The $(1-x)\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ nanoparticles were used as catalysts in wastewater during a photocatalytic process. Orange G was chosen as an impurity in the water. The absorbance curves of Orange G in the water were measured at different times in the presence of the same quantity of $\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ NPs. After 120 min, the efficiency reached 13% and 38% for $(1-x)\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ NPs with $x = 0.1$ and 0.7 , respectively. The experimental results were analyzed by pseudo first- and second-order models and intra-particle diffusion models. A first-order kinetic model had the best fit for the $(1-x)\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ NPs. The strongest relationship occurred with a composition of $0.1\text{Cr}_2\text{O}_3/0.7\text{K}_2\text{O}$.

Keywords: Cr_2O_3 ; K_2O ; HR-TEM; Photocatalysis; Optical Properties; Dye pollution

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44 **1. Introduction**

45 Organic pollution is becoming a health issue worldwide. In particular, dyes are
46 discharged by many dye industries and homes through wastewater into the environment. Research
47 is now being conducted to develop semiconductor photocatalysts that can be used to remedy this
48 damage through water disinfection, hazardous waste remediation, and water purification
49 (Fujishima et al., 2000; Trawiński and Skibiński, 2019; Peral and Ollis, 1992). Metal oxides are
50 important in environmental science, electrochemistry, biology, chemical sensors, magnetics, and
51 other fields. Chromium oxide (Cr_2O_3) is one of the most important transition metal oxides and is
52 widely used in many industries as a semiconductor photocatalyst. Chromium oxide is a p-type
53 semiconductor with wide band gap energy (~ 3 eV) (Singh et al., 2019). It also possesses excellent
54 optical and electrical properties, is low cost, chemically stable, nontoxic, and has surface properties
55 that affect its usage as an industrial catalyst in many reactions (Salaeva et al., 2019; El-Shobaky et
56 al., 2011; Jibril, 2004) Chromium has different stable oxidation states hence the formation and
57 properties of chromium oxide (Cr_2O_3) are important for applied applications such as in high-
58 temperature resistant materials (Jaswal et al., 2014) corrosive resistant materials (Li et al., 2001),
59 liquid crystal displays (Jaswal et al., 2014; Subhan et al., 2019; Freemantle, 1998), green pigment
60 (Fasick et al., 1999) and catalysts (Rotter et al., 2004; Mei et al., 2019). The most effective method
61 to enhance and control the electrical and optical properties of metal oxides is to mix them with
62 selective elements (Viswanatha et al., 2012). However, the preparation of $\text{Cr}_2\text{O}_3/\text{K}_2\text{O}$ catalysts has
63 not been studied. The aim of this study is to prepare $(1-x)\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ nanomaterials with different
64 K_2O contents. Pure K_2O mixed Cr_2O_3 nanoparticles were synthesized through combustion. The
65 structural and optical characterizations of these nanoparticles were characterized by X-ray
66 diffraction (XRD), transmission electron microscopy (TEM), and UV-Vis spectroscopy
67 techniques. Finally, applications of the synthesized nanomaterials as photocatalysts for the
68 degradation of organic Gs (Orange G) were studied. The effect of K_2O mixed Cr_2O_3 in different
69 concentrations, quantities, and irradiation time on the photocatalytic performance was examined.

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2. Materials and Methods

2.1 Dyes

The structure of Orange G dye ($C_{16}H_{10}N_2Na_2O_7S_2$), a crystal orange color anionic dye, (C.I.16230, m.w. 452.37 g/mol) is illustrated in **Fig. 1**.

2.2 K_2O/Cr_2O_3 nanocomposite preparation

Combustion was used to prepare Cr_2O_3 NPs (Rashad et al., 2019; Shaalan et al., 2016; Rashad et al., 2018). 25 ml of a water solution containing $0.1-MCr(NO_3)_2 \cdot 6H_2O$ was mixed with 25 ml of a water solution containing $0.1-MCO(NH_2)_2 \cdot 6H_2O$. Then the mixture was heated for 20 min in a 650-W microwave oven. Cr_2O_3 NPs of green powder were extracted. The procedure was used for preparing K_2O NPs using $0.1-M K(NO_3)_2 \cdot 6H_2O$ as a starting material. A mixture of Cr_2O_3/K_2O powder was prepared with different concentrations of $(1-x)Cr_2O_3/xK_2O$ where ($x = 0.1, 0.3, 0.5$, and 0.7).

2.3 Characterizations

$CuK\alpha$ radiation ($\lambda = 1.546 \text{ \AA}$) was used in the X-ray (Philips PW 1710 diffractometer) to study the crystal structure at temperatures between 5 and 90 °C. JCPDS-ICDD was used to analyze the XRD phases. High-resolution transition electron microscopy (HR-TEM, JEM-2100) was used for analyzing the images of the prepared nanoparticles. Additionally, scanning image observation was used for light and dark-field TEM at 200 kV. Absorption (Perkin Elmer 750 Lambda) was used for the optical measurements at wavelengths from 200 to 900 nm.

The photocatalytic effect was illustrated by Orange G degradation rate using $(1-x)Cr_2O_3/xK_2O$ NPs with ($x = 0.1, 0.3, 0.5$, and 0.7). NPs as a catalyst. 0.02 mg of these nanoparticles was added to 4 ml of Orange G solution (8 mg/L). The solution was added in ultrasonic instrument for 10 min then transfer to glass tubes under ultraviolet lamp with power 6 W separated by 15 cm. The absorbance of the solution Orange G in the presence of the catalysis $(1-x)Cr_2O_3/xK_2O$ NPs with ($x = 0.1, 0.3, 0.5$, and 0.7) was measured in the range of time (0–120) min.

3 Results and Discussion

3.1 Structural characterization

Fig. 2 depicts the XRD patterns of (1-x)Cr₂O₃/xK₂O NPs with (x = 0.1, 0.3, 0.5, and 0.7). The XRD analysis pattern of the (1-x)Cr₂O₃/xK₂O nanocomposite is in good agreement with standard JCPDS No. 00-026-1327 (K₂O) and 04-021-0178 (Cr₂O₃), confirming the existence of two phases in the single matrix. The observed diffraction peaks were monoclinic structures of Cr₂O₃ and hexagonal structures of K₂O. Several observations with increasing K₂O content can be illustrated with XRD charts. First, there is a shift in peaks (-112), (-204), (020), (-113), and (-316) to small diffraction angles, indicating the incorporation of K₂O with the Cr₂O₃ matrix. Secondly, by adding K₂O with varying concentrations, diffraction peaks related to the K₂O phase were observed at $2\theta \approx 25.08^\circ, 27.07^\circ, 29.08^\circ, 29.72^\circ, 31.23^\circ$, and 38.76° . Lastly, the figure confirms that the majority of the mixture is composed of Cr₂O₃.

The size and strain of the Cr₂O₃ crystals were estimated using the Scherrer formula (Shaaban et al., 2019):

$$D = \frac{0.9\lambda}{B \cos\theta} \quad (1)$$

$$\varepsilon = \frac{\beta}{4 \tan \theta} \quad (2)$$

where D = crystalline size, λ = radiation wavelength, B = full width at half maximum at diffraction angle 2θ . Celref UNITCELL software was used to calculate lattice parameters from the XRD data. The calculated lattice parameters and d-spacing of Cr₂O₃ were compared to the standard data recorded in card No. 04-021-0178 and listed in **Table 1**. From this table, it can be seen that the average crystalline size decreased with increasing K₂O concentration from 33.54 nm to 28.37 nm.

The TEM images of (1-x)Cr₂O₃/xK₂O where x = 0.1 and 0.7 are shown in **Fig. 3**. The TEM observations provide information about shape and size. It was found that the average diameter of (1-x)Cr₂O₃/x K₂O NPs where x = 0.1 and 0.7 is ~ 100 nm and 70 nm, respectively. Different shapes such as irregularly shaped and hexagonally shaped nanoparticles are also visible. The inset electron diffraction pattern (SAED) taken from the selected Cr₂O₃ NPs confirms the XRD pattern.

3.2 Optical spectra of the nanoparticles

The changes in optical absorption spectra of (1-x)Cr₂O₃/xK₂O where (x = 0.1, 0.3, 0.5, and 0.7) are shown in **Fig. 4**. The absorption edge of the (1-x)Cr₂O₃/xK₂O samples varies with the varying content of K₂O in the Cr₂O₃ nanoparticles. As K₂O content increases, the blue shift of the optical band gap can be clearly observed. The blue shift indicates a minor modification in the band

structure when mixing $\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$. The absorption coefficient α was calculated for each wavelength using the following equation (Moharram et al., 2014):

$$\alpha = \frac{2.3A}{t} \quad (3)$$

where A is the absorbance equal to $\log I_0/I$ (according to Beer-Lambert's law), $t = 1$ cm is the path length, and I_0 and I are the intensity of incident and transmitted light, respectively (Moharram et al., 2014).

The dependence of absorption spectra on the optical band gap energy (E_g) was described by Tauc's expression (Al-Aoh et al., 2013):

$$\alpha h\nu = \beta(h\nu - E_g)^r \quad (4)$$

where β is a constant and $r = 1/2$ for direct transition due to photon absorption. The plot of $(\alpha h\nu)^2$ versus $h\nu$ shown in **Fig. 5** is linear at the absorption edge, indicating a direct transition. The energy gap is determined by extrapolating the straight line portion of the energy axis at $(\alpha h\nu)^2 = 0$. It is observed that the resultant values of E_g range from 2.40 to 2.61 eV as the K_2O content increases from $x = 0.1$ to $x = 0.7$.

3.3 Photocatalysis applications of $\text{Cr}_2\text{O}_3/\text{K}_2\text{O}$ NPs

Photocatalytic debasement is a procedure for disposing of both natural and inorganic contaminants in waste water where distinctive nanoparticles are utilized to eliminate these contaminants. Metal oxides are a particularly effective catalyst for mitigation (Lin et al., 2015; Rashad et al., 2019) and blended nanoparticles could have an even greater efficacy. Therefore, $(1-x)\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ NPs where ($x = 0.1, 0.3, 0.5$, and 0.7) were tested as catalysts with varying contents of K_2O NPs. **Fig. 6** shows Orange G absorption spectra for $(1-x)\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ NPs where ($x = 0.1, 0.3, 0.5$, and 0.7) at various exposure times. It can be seen that the absorbance of Orange G is distinguished by one peak at 480 nm. The absorbance spectra show that Orange G was diminishing as the UV light time increments for each $(1-x)\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ NP where ($x = 0.1, 0.3, 0.5$, and 0.7). To quantify the impact of K_2O content (x), the photocatalytic productivity (η) of Orange G was determined with the equation (Zhu et al., 2012):

$$\eta\% = \frac{C_o - C_t}{C_o} \times 100 = \frac{A_o - A_t}{A_o} \times 100 \quad (5)$$

where C_o = initial Orange G concentration, C_t = residual Orange G concentration after the specified time, A_o = initial Orange G absorbance, and A_t = residual Orange G absorbance after the specified

time. **Fig. 7** shows the photocatalytic efficiency of Orange G with $(1-x)\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ NPs where ($x = 0.1, 0.3, 0.5, \text{ and } 0.7$). Photocatalytic degradation occurred with a maximum of 35.6% after irradiation time of 120 min at $x = 0.7$. The photocatalytic process (Rashad, 2019) is explained as a photo-induced molecular transformation occurring at the surface of the metal oxide NP. The electron-hole pair is generated when light is utilized in the photocatalytic process. When the catalyst is exposed to UV light with higher energy than its band gap, the electron is transported to the conduction band creating a hole in the valence band. These holes are important for photocatalysis because they are a source of oxidized hydroxyl radicals that react to the electrons in the dyes (Rashad et al., 2016; Curri et al., 2003). See sketch 1.

The following equations describe the previous procedures (Al-Aoh, 2018):

3.4 Kinetic analysis

The mass of Orange G degraded per unit of $(1-x)\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ NPs (q , mg/g) at a fixed time and equilibrium state were calculated using the following equations (Ahmed et al., 2011):

$$q_t = (C_i - C_t) \frac{V}{W} \quad (11)$$

$$q_e = (C_i - C_e) \frac{V}{W} \quad (12)$$

where q_t = the quantity of degraded Orange G at time (t) in mg/g, q_e = the quantity of degraded Orange G at equilibrium in mg/g, C_i = the initial concentration of Orange G solution in mg/L, C_t = the concentration of degraded Orange G at time (t) in mg/L, C_e = the concentration of degraded Orange G at equilibrium in mg/L, V = the volume of the solution in L, and W = mass in g.

The relationship of degraded Orange G, q_t , with the amount of light for $(1-x)\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ NPs where ($x = 0.1, 0.3, 0.5, \text{ and } 0.7$) is shown in **Fig. 8**. These curves show that as the illumination time increases, the q_t values increase for all compositions of $(1-x)\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ NPs. The impact of K_2O NP content increases the degraded Orange G capacity, confirming the photocatalytic procedure (Darwish et al., 2019). Pseudo first-order, pseudo second-order, and intra-particle diffusion kinetic models were utilized (Rashad and Al-Aoh, 2019) for analysis:

$$\log(q_e - q_t) = \log q_e - K_1 \frac{t}{2.303} \quad \text{Pseudo First-Order Model} \quad (13)$$

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e} \quad \text{Pseudo Second-Order Model} \quad (14)$$

$$q_t = K_{diff} \sqrt{t} + C \quad \text{Intra-particle Diffusion} \quad (15)$$

where K_1 = the pseudo first-order constant in g/mg·min, K_2 = the pseudo second-order constant in g/mg·min, K_{diff} = the intra-particle diffusion kinetic model in (mg/g·min)^{1/2}, and C = the kinetic parameter constant.

The models described by Eqs. 9, 10, and 11 can be utilized to clarify the experimental Orange G degradation onto the surface of (1-x)Cr₂O₃/xK₂O NPs where ($x = 0.1, 0.3, 0.5$, and 0.7). The pseudo first-order model for Orange G debasement is described by Eq. 9. The dependence of $\log(q_e - q_t)$ on t for (1-x)Cr₂O₃/xK₂O NPs where ($x = 0.1, 0.3, 0.5$, and 0.7) is illustrated in **Fig. 9**. The linear fit provides the kinetic parameter estimations of K_1 , determined q_e , and the coefficient (R^2), which are recorded in **Table 2**. The second-order model for Orange G degradation is displayed by Eq. 10. The t/q_t variable is dependent on t as shown in **Fig. 10**. The linear fit is used to calculate K_2 , q_e , and the coefficient (R^2), which is recorded in **Table 1**. It is inferred that the estimation of R^2 for the pseudo first-order is higher than the estimate for the pseudo second-order. In addition, the distinction between the calculated q_e and experimental q_e for the pseudo first-order is less than that of the pseudo second-order model. Accordingly, the pseudo first-order model is progressive for delineating the Orange G degradation using (1-x)Cr₂O₃/xK₂O NPs where ($x = 0.1, 0.3, 0.5$, and 0.7). Intra-particle diffusion parameters were determined by Eq. 11, which demonstrates the reliance of q_t on $\sqrt{t}$ as represented in **Fig. 11**. The linear fit provides estimates of K_{diff} and C for (1-x)Cr₂O₃/xK₂O NPs where ($x = 0.1, 0.3, 0.5$, and 0.7). It can be seen that Orange G debasement has occurred in a single step, which implies that the Orange G degradation is a chemisorption process. These results are comparable with previous data (Rashad, 2019; Al-Aoh, 2013).

Conclusions

XRD, HR-TEM, and optical measurements were used to investigate nanoparticles of $(1-x)\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ where ($x = 0.1, 0.3, 0.5$, and 0.7). These measurements provide a complete overview of the structure, shape, and optical properties of the nanoparticles. Both photocatalytic efficiency and kinetic parameters of Orange G in wastewater were investigated using $(1-x)\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ NPs where ($x = 0.1, 0.3, 0.5$, and 0.7). The efficiency obtained with $(1-x)\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ where $x = 0.1$ and 0.7 was 13% and 38%, respectively. These photocatalytic and kinetic studies confirm that Orange G degradation with catalysts of $\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ NPs is through chemisorption.

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311 **Table 1:** Structural properties of (1-x)Cr₂O₃/xK₂O nanoparticles

<i>x</i>	<i>Average crystallite size (nm)</i>	<i>Lattice parameter [Å] (a,b,c)</i>	<i>Unit cell volume (Å³)</i>	<i>Micro strain (ε)</i>
0.1	33.54	7.2974 4.9486 5.3495	192.04	0.003415
0.3	32.88	7.2914 4.9549 5.3506	192.22	0.003443
0.5	28.81	7.3009 4.9605 5.3605	193.04	0.003972
0.7	28.37	7.2928 4.9700 5.3540	192.97	0.003996

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313 **Table 2:** Pseudo first-, pseudo second- order parameters and experimental q_e and parameter values
314 of intraparticle diffusion model for the Orange G using (1-x)Cr₂O₃/xK₂O (x=0.1, 0.3, 0.5, 0.7) NPs.

<i>x</i>		<i>x</i> =0.1	<i>x</i> =0.3	<i>x</i> =0.5	<i>x</i> =0.7
Experimental q _e	q _e (exp.) (mg/g)	124	135	224	257
Pseudo first-order kinetic model	q _e (mg/g)	144	113	251	309
	Δq _e (first-order) = q _e (exp.)-q _e	20	22	27	52
	K ₁ (h ⁻¹)	0.032	0.012	0.020	0.030
	R ²	0.80	0.80	0.80	0.95
Pseudo second-order kinetic model	q _e (mg/g)	125	100	200	273
	Δq _e (second-order) = q _e (exp.)-q _e	1	22	24	16
	K ₂ (g/mg.min)	8.5x10 ⁻⁴	2.5 x10 ⁻³	2.1x10 ⁻⁴	1.4 x10 ⁻⁴
	R ²	0.86	0.98	0.55	0.50
Intra-particle diffusion kinetic model	K _{eff} (mg/h ^{1/2} g)	12.4	10.12	17.9	25.3
	C	2.05	5.6	-9.7	-13.6
	R ²	0.95	0.93	0.90	0.91

Fig. 1: Orange G chemical structure

Sketch 1: Photo catalysis mechanism

Fig. 2: XRD patterns of $(1-x)\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ NPs. prepared in different percentages ($x = 0.1, 0.3, 0.5, 0.7$).

Fig. 3: Typical HRTEM images of $(1-x)\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ where ($x = 0.1$ and 0.7)NPs

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338 **Fig. 4:** UV-Vis Spectrum of $(1-x)\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ ($x=0.1, 0.3, 0.5, 0.7$) NPs.

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Fig. 5: Plot of $(\alpha h\nu)^2$ vs photon energy ($h\nu$) for different concentration of $(1-x)\text{Cr}_2\text{O}_3/x \text{K}_2\text{O}$ NPs. where ($x=0.1, 0.3, 0.5, 0.7$).

Fig. 6: Absorbance time dependence of Orange G with $(1-x)\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ ($x=0.1, 0.3, 0.5, 0.7$) as catalyst

Fig. 7: The photocatalytic efficiency ($\eta\%$) of Orange G debasement using $(1-x)\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ ($x=0.1, 0.3, 0.5, 0.7$) NPs.

Fig. 8: Degradation capacity (q_t) of Orange G versus illumination time on the $(1-x)\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ ($x=0.1, 0.3, 0.5, 0.7$) NPs.

Fig. 9: Pseudo first-order kinetic model for Orange G using the $(1-x)\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ ($x=0.1, 0.3, 0.5, 0.7$) NPs.

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368 **Fig. 10:** Pseudo second-order kinetic model for Orange G using the $(1-x)\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ (x=0.1, 0.3,
369 0.5, 0.7) NPs.

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371 **Fig. 11:** Intra-particle diffusion model for Orange G using $(1-x)\text{Cr}_2\text{O}_3/x\text{K}_2\text{O}$ (x=0.1, 0.3, 0.5, 0.7)
372 NPs.