Data-Driven Gas Sensing Analysis of Ti-Doped Cdo Thin Films

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Keywords: Cdo, Ti, Thin Films, Doping, Physical Properties, Spray Pyrolysis.

Abstract: This work examines how titanium doping modifies the structural characteristics, surface morphology, and optical behavior of nanostructured CdO films fabricated through chemical spray pyrolysis (CSP). Thin films of pure CdO and Ti-incorporated CdO (at 2% and 4% doping levels) were successfully fabricated under controlled conditions, with precise temperature regulation at 450°C and uniform thickness distribution of 330±20 nm. XRD characterization revealed that all films exhibited a crystalline structure with predominant (111) orientation. The Grain size increased from 14.15 nm (undoped) to 17.83 nm (4% Ti-doped), while dislocation density and microstrain showed significant reduction, confirming enhanced crystalline perfection with Ti incorporation. AFM analysis revealed that Ti doping notably altered the surface morphology, reducing the average grain size by 46% (from 72.74 nm to 39.46 nm) and RMS roughness by 63% (from 8.76 nm to 3.21 nm), leading to enhanced surface uniformity. Optical characterization revealed an inverse relationship between Ti concentration and light transmittance, with concurrent increases in both absorption coefficient (α) and extinction coefficient (k). Ti-doped CdO sensors, especially at 4%, showed increased resistance and sensitivity to NO_2 due to enhanced surface reactivity and electronic modification. With increasing Ti concentration, CdO sensor sensitivity to NO_2 decreases significantly, dropping across all tested ppm levels due to reduced surface-to-volume ratio and increased charge carrier recombination.

1 INTRODUCTION

In the past decade (2014-2024), cadmium oxide (CdO) has emerged as a high-performance transparent conducting oxide (TCO), attracting significant research attention optical transparency (exceeding 80% in the visible range) and low electrical resistivity (below 10^{-3} Ω·cm). This dual functionality makes CdO a promising material for a wide range of advanced optoelectronic applications, including next-generation photovoltaic devices, ultra-sensitive gas sensors, and energy-efficient optoelectronic systems [1]-[4]. (CdO) is an n-type semiconductor, naturally demonstrates n-type degenerate semiconducting properties [5], [6]. Controlled elemental doping in such metal oxide semiconductors serves as an essential mechanism for tailoring both optical and electrical properties, enabling optimized performance in photovoltaic devices [7]. Doping CdO with selected elements (F, Mn, Mo, Ti, Sn) enables precise modification of its optoelectronic properties. The incorporated dopants increase carrier concentration and conductivity, resulting in a Burstein-Moss mediated bandgap blue shift [8]. This unique combination of high visible-range transparency (T > 80%) with excellent electrical properties (ρ < 10-3 Ω·cm) makes CdO particularly valuable for transparent electronics [9]. Researchers have developed multiple fabrication techniques for CdO thin films, including solution-processed sol-gel methods [10], chemical bath deposition [11], SILAR [12]-[14], CSP [15]-[17], CVD [18], PLD [19], [20], vacuum evaporation [21] and magnetron sputtering [22]-[24]. Among various thin film deposition methods, spray pyrolysis stands out as a cost-effective and straightforward chemical approach capable of producing large-area thin films [5]. Modern spray pyrolysis techniques enable controlled doping of semiconductor thin films, allowing accurate incorporation of multiple dopant species at predetermined concentrations via optimized precursor chemistry [18]. This study aims to achieve two main objectives: first, the development of CdO films using CSP technique; and second, a systematic investigation of the physical properties of the entended films.

2 EXPERIMENTAL

CdO thin films were fabricated using CSP technique, utilizing an aqueous precursor solution prepared by dissolving 0.1 M of ($CdOCl_2 \cdot 8H_2O$, BDH Chemicals) and Oxalic acid (from Merck Chemicals) was dissolved in 100 mL of deionized water. The solution was thoroughly stirred to ensure complete dissolution and uniform distribution of the precursors before the deposition process. Titanium doping was achieved using 0.1 M titanium(IV) chloride tetrahydrate ($TiCl_2 \cdot 4H_2O$) as the dopant source, with concentrations of 2% and 4% relative to cadmium to produce Ti-doped CdO films. During the deposition process, the base temperature was consistently maintained at 450°C. Through systematic optimization, we established the following spray parameters: nozzle-to-substrate distance (29 cm), spray duration (9 seconds), solution flow rate (5 mL/min), and spray interval (90 seconds between cycles). High-purity nitrogen served as the carrier gas during deposition. Film thickness, determined gravimetrically, averaged 330 ± 20 nm. The crystalline structure and preferred growth orientation were analyzed using XRD, with results benchmarked against ASTM reference standards. Surface morphology analysis including grain size and roughness measurements was performed using atomic force microscopy (NT-MDT SPM Integra AFM). Optical properties were characterized through UV-Vis spectroscopy (300-900 nm range) measuring both transmittance and absorption spectra with a double-beam spectrophotometer. Gas sensitivity is typically measured by the percentage change in the film's resistance upon gas exposure. The samples were tested inside a custom-made cylindrical chamber measuring 9 cm in radius and 17 cm in height.

3 RESULTS AND DISCUSSIONS

The X-ray diffraction (XRD) patterns of pure CdO and Ti-doped CdO films (2% and 4%) are presented in Figure 1. All samples exhibit four well-defined diffraction peaks at 37.38°, 38.47°, 55.57°, and 66.07°, corresponding to the (111), (200), (220), and (311) planes of the cubic CdO crystal structure, consistent with JCPDS card 05-0640 [25], [26]. The most intense peak is observed at (111), indicating a preferred orientation of crystal growth along this direction and confirming the polycrystalline nature of the deposited films. In addition, the absence of any noticeable peak shift with increasing Ti concentration suggests that Ti incorporation does not alter the crystal symmetry or induce secondary phases, indicating successful substitutional doping without significant lattice distortion [27], [28].

The average crystallite size was estimated using Scherrer's approach [29], [30], where parameters such as X-ray wavelength (Cu Kα radiation, 0.15406 nm), shape factor (0.9 for near-spherical grains), full width at half maximum, and Bragg angle were considered. The obtained results show that the crystallite size increases progressively from 14.15 nm for the undoped sample to 17.83 nm for the film doped with 4% Ti, corresponding to an overall growth of about 26%. This trend suggests that Ti incorporation enhances crystal coalescence, reduces grain boundary density, and may facilitate crystallization during the spray pyrolysis process [31], [32].

The dislocation density, which reflects the level of crystalline defects, was evaluated as described in [33], [34]. A clear reduction is observed with doping, decreasing from 53.4×10^{14} lines/m^2 in the pure CdO film to 20.8×10^{14} lines/m^2 at 4% Ti concentration, which represents a significant reduction of approximately 61% [35], [36]. This behavior indicates that Ti doping effectively reduces structural imperfections, supports more ordered growth, and minimizes internal strain within the lattice [37], [38].

Lattice microstrain was assessed using the Williamson-Hall method [39], [40]. The results demonstrate a decrease in microstrain values with increasing Ti content, dropping from 26.47×10^{-4} in undoped CdO to 17.35×10^{-4} for the 4% doped sample, corresponding to a reduction of about 34.5% [41]. This suggests that Ti incorporation relieves internal stresses, promotes relaxed crystal formation, and compensates for native defects within the CdO structure [42].

Figure 2 summarizes all calculated structural parameters, while Table 2 lists the corresponding numerical values. Figure 3 presents AFM images of both pure and Ti-doped CdO films, showing notable changes in surface morphology. The average particle size decreases from 72.74 nm for the undoped film to 50.60 nm at 2% Ti and further to 39.46 nm at 4% Ti, indicating a substantial refinement of surface grains by nearly 46% with doping. In parallel, surface roughness parameters (RMS roughness and average roughness) also decrease significantly, confirming a smoother and more uniform surface. These observations demonstrate that Ti incorporation improves microstructural uniformity and enhances surface quality [43]. Table 1 summarizes the AFM-derived parameters of all films.

Figure 4 displays the optical transmittance spectra of the investigated films. The undoped CdO sample shows a maximum transparency of about 80% at 550 nm. With 2% Ti doping, this value decreases to approximately 70%, and further reduces to around 60% at 4% doping, representing an overall reduction of roughly 25% in the visible region. This decrease is mainly attributed to increased free carrier absorption associated with the Drude mechanism and enhanced light scattering at grain boundaries [44].

Absorbance values were derived from the transmittance spectra using the standard logarithmic relationship [45], [46].

Figure 5 illustrates A spectra of both pure and Ti-doped CdO thin films. As the titanium doping concentration increases, the absorption edge shifts from around 300 nm to 500 nm, indicating a red shift and corresponding reduction in the band gap. This shift is attributed to the incorporation of Ti atoms into the CdO lattice, which introduces localized energy states near the conduction band and alters the electronic structure. Additionally, the absorption intensity increases with higher Ti doping levels, which can be explained by enhanced free carrier concentration, increased defect density, and stronger electron-photon interactions, all of which contribute to greater light absorption across the UV-visible region [47].

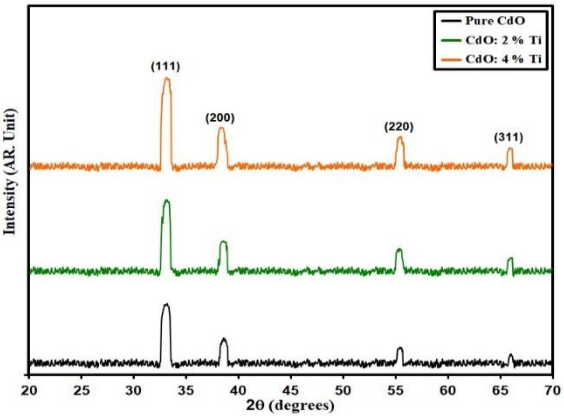

Figure 1: XRD patterns of the grown films.

Table 1: P_{AFM} of the deposited films.

Samples	P_{av} (nm)	Ra (nm)	RMS (nm)
Pure CdO	72.74	7.88	8.76
CdO: 2% Ti	50.60	6.63	4.45
CdO: 4% Ti	39.46	3.21	3.21

Table 2: D, E_g, and SP of the deposited films.

Sample	(hkl) Plane	2 θ (°)	FWHM (°)	E_g (eV)	D (nm)	δ (× 10^{14}) (lines/m^2)	ε × 10^{-4}
Pure CdO	111	33.38	0.61	2.77	14.15	53.4	26.47
CdO: 2% Ti	111	33.34	0.56	2.72	16.93	41.9	20.19
CdO: 4% Ti	111	33.30	0.52	2.68	17.83	20.8	17.35

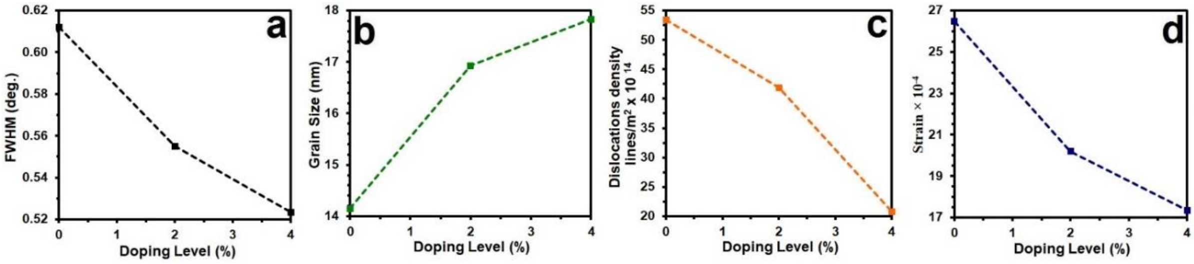

Figure 2: SP vs. doping of deposit films.

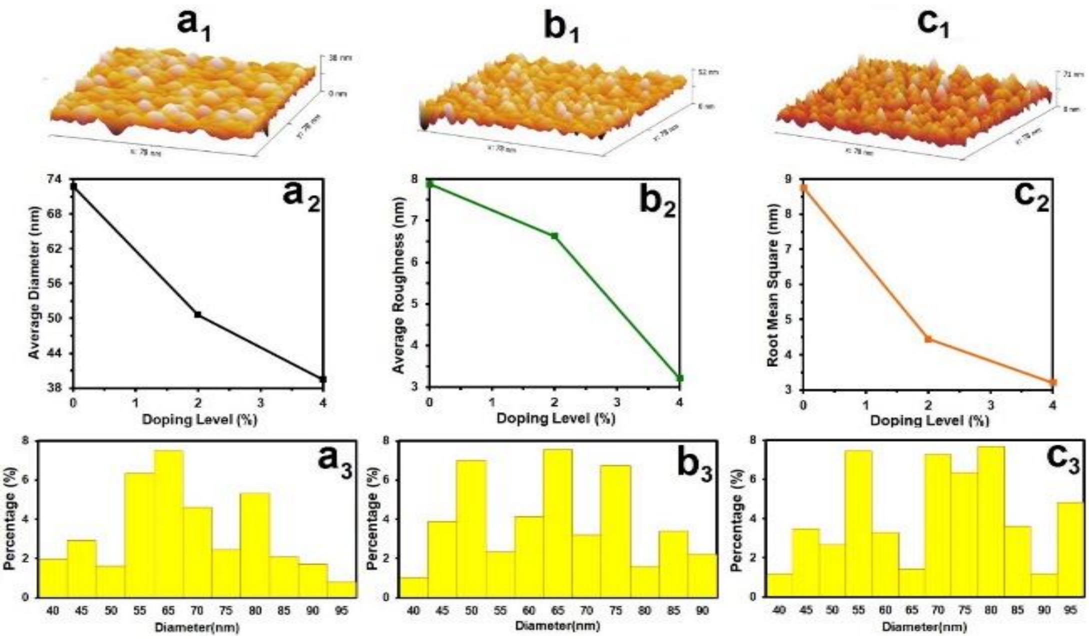

Figure 3: AFM surface morphology of pure and Ti-doped CdO thin films.

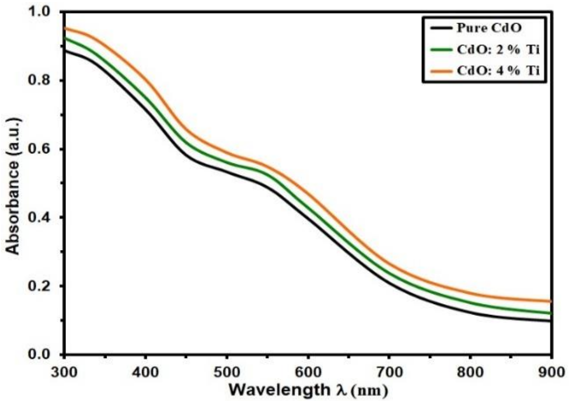

Figure 4: The absorbance spectra of the deposit films.

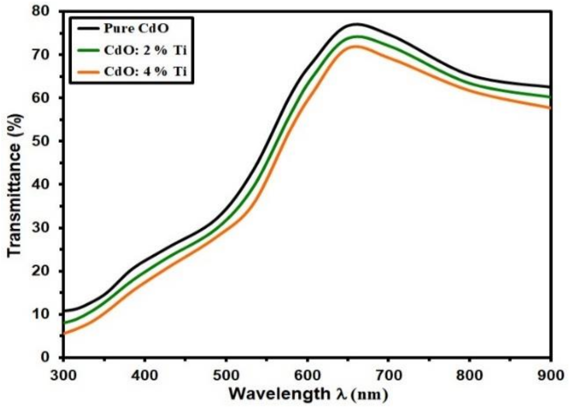

Figure 5: T of the deposit films.

The absorption coefficient (α) was determined using a standard logarithmic relation based on the measured transmittance data [48], [49], where film thickness is taken into account. The obtained values of α are in the range of 10^4 cm^{-1}, indicating strong absorption characteristics of the CdO thin films. As illustrated in Figure 6, the absorption coefficient increases with photon energy, confirming a high probability of optical transitions at elevated energies. This trend is typical for direct bandgap semiconductors, where absorption becomes more efficient as photon energy rises.

In addition, an increase in titanium concentration leads to a noticeable enhancement in absorption, particularly near the absorption edge. This behavior is associated with the formation of localized energy states and increased free carrier contributions introduced by Ti doping, which modifies the electronic structure and strengthens the interaction between light and the material [50].

The optical bandgap (Eg) was estimated using the Tauc method for direct allowed transitions [51], [52], as shown in the corresponding analysis. The results in Figure 7 demonstrate a gradual decrease in bandgap energy with increasing Ti content. Specifically, Eg decreases from 2.77 eV for undoped CdO to 2.68 eV for the film doped with 4% Ti [53]. This reduction in bandgap energy is attributed to the Burstein–Moss effect, which arises from changes in carrier concentration induced by Ti incorporation.

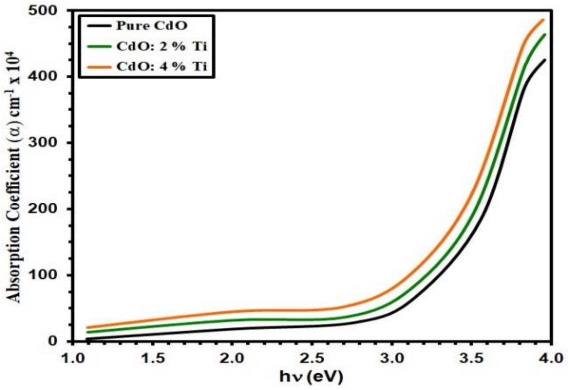

Figure 6: α Vs hν for the prepared films.

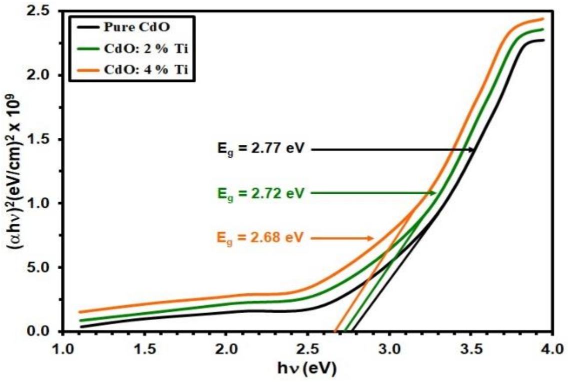

Figure 7: $(\alpha h\nu)^2$ Vs hν for the prepared films.

The extinction coefficient (k) was obtained from the absorption coefficient using the standard optical relation [54], [55]. The calculated values show that k decreases with increasing wavelength for all Ti-doped CdO thin films, as presented in Figure 8. Moreover, a clear reduction in k is observed with higher Ti concentrations. This trend may be explained by the influence of Ti incorporation on the microstructure and electronic properties of the films, which can reduce free carrier concentration and modify light–matter interaction processes, resulting in lower absorption and scattering losses [56].

The refractive index (n), which depends on wavelength, was evaluated using the established optical expression that incorporates both reflectance and extinction coefficient [57], [58]. The behavior of n as a function of wavelength is shown in Figure 9 for pure and Ti-doped CdO films. It is evident that the refractive index decreases with increasing Ti content [59]. This reduction is likely related to structural modifications induced by Ti incorporation, such as decreased packing density and increased porosity, which disrupt the regular crystal arrangement and reduce overall optical density.

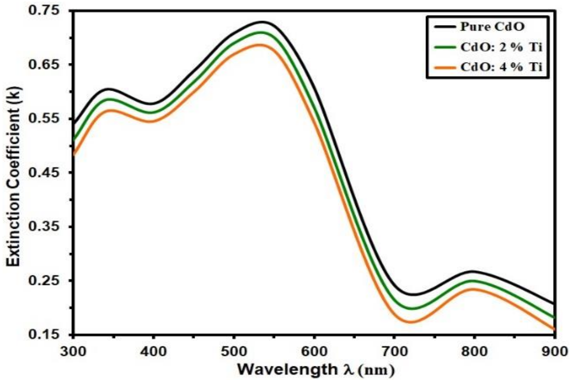

Figure 8: k of the grown films.

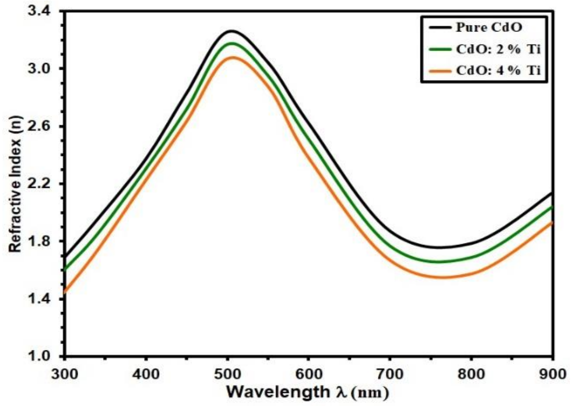

Figure 9: n of the enteded films.

The gas sensing performance was assessed using a standard sensitivity definition based on the relative change in electrical resistance [60], [61]. The CdO-based sensors, fabricated on porous silicon substrates and coated with pure and Ti-doped (2% and 4%) CdO films, were tested for NO_2 detection at 240 ppm and an operating temperature of 90 °C. Figure 10 presents the time-dependent resistance response of the sensors. Upon exposure to NO_2, surface oxidation processes occur, where adsorbed oxygen species release trapped electrons back into the conduction band, leading to an increase in resistance due to enhanced grain boundary potential barriers [62], [63]. Among all samples, the film doped with 4% Ti exhibits the highest resistance response, indicating improved sensing activity, which can be attributed to enhanced surface reactivity and electronic modifications caused by Ti incorporation [64].

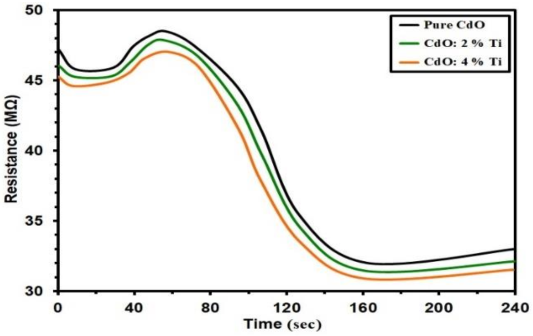

Figure 10: Resistance via time for the deposit films.

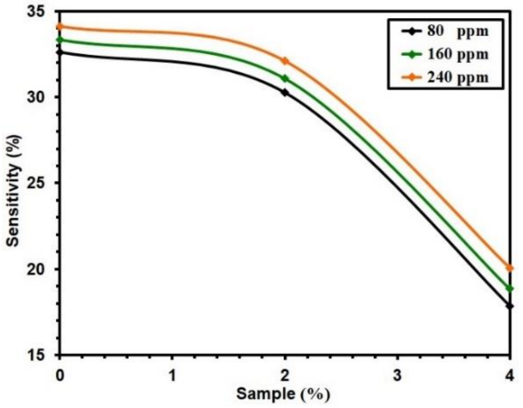

Figure 11: sensitivity (S) as a function of operating time for of Pure CdO and CdO: % Ti thin film.

The sensitivity results, shown in Figure 11, further demonstrate the influence of Ti doping on NO_2 detection performance. A gradual decrease in sensitivity is observed with increasing Ti content, which is mainly related to charge carrier recombination effects and reduced active surface area. Specifically, the sensitivity decreases from 32.6% to 17.8% at 80 ppm, from 33.3% to 18.86% at 160 ppm, and from 34.1% to 20.0% at 240 ppm for pure, 2% Ti-doped, and 4% Ti-doped CdO films, respectively [65]. This reduction is attributed to the decrease in surface-to-volume ratio caused by higher Ti doping levels, which limits the number of available active sites for gas adsorption and consequently weakens the overall sensor response [66].

4 CONCLUSIONS

This systematic study demonstrated titanium doping's significant influence on CdO:2% and CdO: 4% Ti thin films, successfully modifying their structural, morphological, and optical characteristics through controlled Ti incorporation. XRS analysis confirmed the polycrystalline structure of all deposited films, exhibiting a preferred (111). Ti doping increased crystallite size from 14.15 nm (pure CdO) to 17.83 nm (4% Ti-doped CdO), while reducing dislocation density and strain, indicating improved crystallinity. AFM revealed that Ti doping refined grain size (from 72.74 nm to 39.46 nm) and reduced surface roughness (from 8.76 nm to 3.21 nm). The optical bandgap (Eg) decreased from 2.77 eV (pure CdO) to 2.68 eV (4% Ti-doped CdO). Transmittance decreased with higher Ti concentrations, while absorption coefficient and extinction coefficient increased. The refractive index exhibited a declining trend with Ti doping. The Ti-doped CdO gas sensor showed increased resistance and enhanced sensitivity to NO_2 (240 ppm) at 90 °C, especially at 4% Ti doping. Increasing Ti concentration reduces sensitivity for CdO due to enhanced charge carrier recombination and fewer active surface sites, lowering the sensor's NO_2 adsorption and response.

ACKNOWLEDGMENTS

This research was supported by Mustansiriyah University (www.uomustansiriyah.edu.iq) through its academic research program.

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