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Study of the atomic packing fraction, specific surface area and morphology index of pure titanium dioxide compound and doped with lead TiO2: Pb in different ratios

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Abstract

In this research, additional physical parameters were calculated for previous parameters that were set for samples of pure titanium oxide compound doped with lead in different proportions according to (x = 0.2 - 0.5 - 0.7 - 0.9) in its three phases (anatase, brookite and rutile) by making use of X-ray diffraction diagrams. XRD, where the computational results of the atomic packing fraction PF, the morphology index MI, and the specific surface area S showed the structural changes that occur to the pure powder as a result of doping it with different proportions of lead, so we have shown from the range to which the morphology index values belong that the compound belongs to pure titanium dioxide, and The values of the morphology index varied according to the different phases and the different doping ratios, and rutile had the largest value of MI at 0.2g Pd doping, and when calculating the specific surface area, the anatase phase appeared with the largest surface area compared to the other two phases at 0.7 Pd doping, then we explained the changes of the morphology index varies by parametric both the crystal size D on the one hand and the specific surface area S on the other hand. We also explained and explained the changes that appeared on the values of the atomic packing fraction of the compound after doping It with lead ratios.

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Introduction

The importance of our research comes from the importance of both the powders and the compound used, as the powders have a greater photocatalytic activity than the thin films of titanium dioxide, as well as the use of TiO_2 compound in many and wide applications, as titanium dioxide is a semiconductor used in photocatalysis [10] and in photovoltaic systems and dye-sensitive photocells [7,8], it has a large band gap Eb = 3.2 eV that makes this catalyst effective only in the ultraviolet region. These doping processes are: TiO_2:SiO_2 [11], TiO_2: N [6], TiO_2 : MgO [13], TiO_2: PbO [1,2], TiO_2:Pb [9,4]. TiO_2 also has a refractive index. It is high, which increases with increasing annealing temperature [3]. This compound also enjoys thermal and chemical stability, non-toxicity [22], its wide applications in optics and electronics [21,5], its biological and chemical inertness, and its mechanical strength [24]. The titanium dioxide compound is characterized by three crystalline phases: anatase and rutile (tetragonal crystal system) and brookite (orthorhombic crystal system) as shown in Figure (1).

In this research, we calculated the atomic packing fraction P F, the morphology index MI, and the specific surface area S (m^2/g) of titanium dioxide compound doped with lead in different proportions, which we studied many of its physical properties previously using the X-ray diffraction device (XRD), where the diffraction charts enabled us For pure and lead-doped samples by calculating several physical values, including the values of crystal size D, cell size V, theoretical density ρ (X-ray) and full width at half maximum intensity (FWHM), which we will use in this research to identify new values. New physical properties in order to enrich the investment of this compound in wider areas [25].

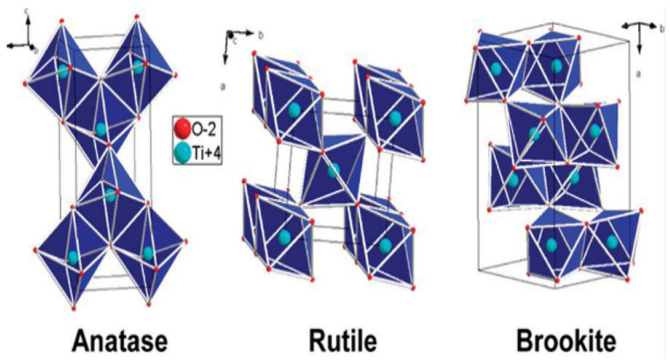

Figure 1: Cystal structure of TiO_2 rutile (tetragonal, $P4_2/mmm$), brookite (orthorhombic, Pbca) and anatase (tetragonal, $I4_1/amd$) polymorphs [18].

Figure 2: Various steps of conventional solid-state reaction method.

Experimental method: The process of preparing samples was carried out in the Faculty of Science at Tishreen University in the laboratories of the Departments of Physics and Chemistry. Pure and lead-doped TiO2 powders in the proportions (x = 0.2 – 0.5 – 0.7 – 0.9) $Ti_{(1-x)}$ Pbx O_2 that we deal with in this research were previously weighed by a sensitive balance type (SARTORIUS) with an accuracy of gr (10-4). Which were manufactured by a solid-state interaction method [20] and [21] and were mixed and grinded well for two hours to become powders using an agate mortar and pestle, very fine, then sifted using a sieve. It has a hole size of 90 micron. Then, all samples were heated up to 200 °C by means of an incinerator.

The aim of the research: Discovering, improving and changing some structural properties of pure titanium dioxide compounds by doping it with metal of lead with different ratios and studying the effect of doping on some of these properties in order to obtain compounds with desirable. Lead was chosen as an impurity because of its distinctive physical properties. Its high density was of great importance in increasing the brookite phase by increasing the percentage of similarity to lead, which was indicated by reference [25]. This is an important conclusion, as the rutile phase is considered the most stable of the three phases and quickly The other two phases transform into it under normal conditions of pressure and temperature.

Results and Calculations

Based on the values of crystal size D, cell size V, theoretical density $\rho_{(X\text{-}ray)}$ and full width at half maximum intensity (FWHM) obtained from the reference study of the XRD plots [25] we were able to calculate the changes that occur To compact the atoms in the compound cell after we doped it with different dopant

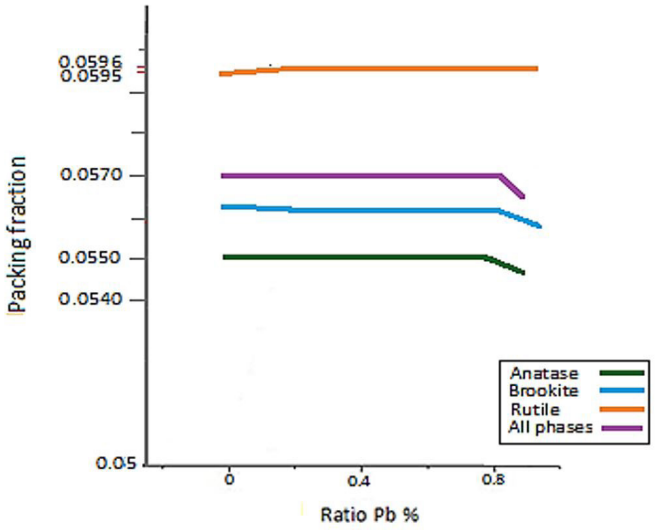

Figure 3: The atomic packing fraction PF of pure and lead-doped titanium dioxide samples at different ratios (x = 0.2 - 0.5 - 0.7 - 0.9) in each of the anatase, brook it and rutile phases, and for both phases. We note that the curves are closer by 99% to the curves that we obtained when drawing the theoretical density changes according to the dopant ratio, and this indicates the existence of a link between the density and the packing fraction, as it increases with the increase in density, and the increase in this fraction may indicate that the crystalline lattice of the samples has a preference In the presence of repetition of the same crystal lattice (symmetry) with glide planes and screw axes that affect the cavities adjacent to it by installing bumps in the single molecular surface [14], where the lowest value of the packing fraction was 0.9, and the phase was the anatase phase, while the highest The value of the packing fraction was for the rutile phase, in which all doping ratios are equal. The packing fraction is evaluated, and the increase in the packing fraction results in an increase in shear stress and an increase in optical conductivity, Therefore, the impediment to the movement of electrons decreases and the generation of electron-hole pairs increases, and this may be an indicator of increased photocatalytic activity, This can be interpreted as a decrease in the distance between two successive crystalline levels, and thus a decrease in the theoretical density and packing fraction [25]. From the figure, the PF maintains a constant value for all pure samples. And the similarity to decrease when the doping ratio is 0.9, due to the increase in cell size and the decrease in the theoretical density. We can say that PF values fall within the range [0.0540-0.0550] for the anatase phase, within the range [0.0595-0.0596] for the rutile phase, within the range for the brookite phase [0.0558-0.0564] and within the range for the whole compound [0.0565-0.0570]. The sample with an impurity ratio of 0.9g was the most different from the pure sample in terms of the values of the PF. Then we calculated the specific surface area S of pure and doped titanium dioxide powders for spherical granules using equation (11), as both equations (10) and (11) give us the same values for the specific surface area [15] and [16], respectively

ratios, we calculated the packing fraction, or the so-called filling fraction, using the relationship (10) [23], which is equal to the ratio of the size of the atoms in the cell to The size of that cell:

$$P.F = \frac{V_P}{V} \qquad (10)$$

P.F: atomic packing fraction

V_P: the size of the atoms present, which is equal to the number of oxygen atoms with the size of one atom $n_1 V_1$ + the number of titanium atoms with the size of one atom $n_2 V_2$.

V: the cell size taken as $(nm)^3$.

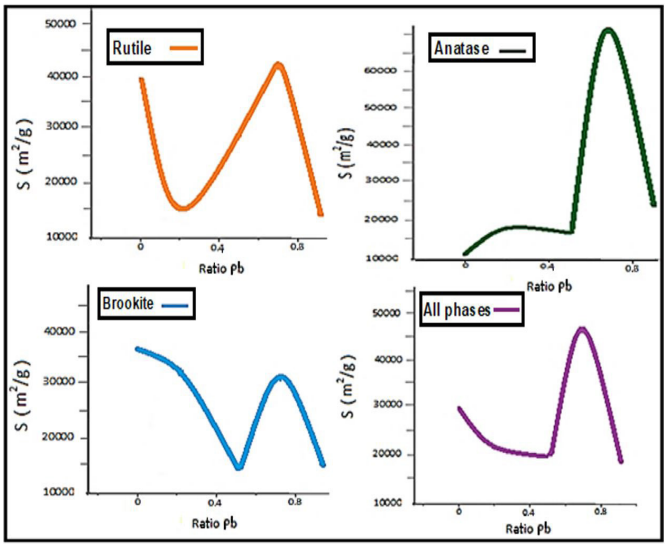

Figure 4: Specific surface area S (m2/g) for pure and lead-doped titanium dioxide samples with different ratios (x = 0.2 – 0.5 – 0.7 – 0.9) in each of the anatase, brookite and rutile phases, and for both phases. All samples are similar in the appearance of the graph in terms of increasing and decreasing the specific surface area at different dopant ratios, except for the anatase sample between the pure compound and the dopant with a ratio of 0.2. The graph shifts between them from decrease to increase. We note that the surface-to-volume ratio decreases with the increase in grain size [12], meaning that the specific surface area decreases with increasing doping ratios, except for the percentage of lead doping that is equal to 0.7, at which the specific surface area increased due to the decrease in the grain size at this ratio only after its increase in the rest. Lead doping ratios, as the study of the specific surface area allows us to identify the possibility of adsorption and non-homogeneous stimulation and the ability of the molecule to move and navigate on the surface and understand the interactions that occur on the surface or at the interface as the adsorption increases with the increase of the specific surface area and thus increases In homogeneous stimulation, among the surfaces of titanium dioxide compounds, it was found that the doping ratio is 0.7. The surface of the compound is active in the adsorption process due to its privilege of not saturating its atoms electronically despite the bonds formed by the atoms of this surface with neighboring atoms [17]. The values obtained are consistent with the researcher [19]. The specific surface area for both phases also fell within the range [47842.78 - 18191.06 m2/g], The anatase phase also had the highest value of the specific surface area compared to the other two phases, which amounted to S = 71061.38 m2/g because the size of its grains was the smallest for the inclusion ratio of g 0.7. S = 12948.71 m2/g, but the predominant phase of the compound, which is the brookite phase, has decreased the values of the specific surface area, giving us as a result a decrease in the values of the latter for the compound for all phases.

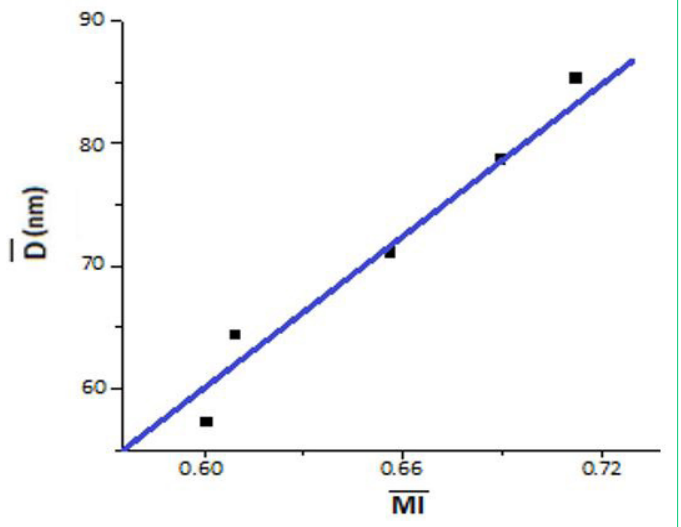

Figure 5: Average crystal size D‾ (nm) of pure and lead-doped titanium dioxide samples with different ratios (x = 0.2 – 0.5 – 0.7 – 0.9) in terms of mean morphology index(MI ‾).

Considering the volume of an atom equals the volume of a sphere:

$$P.F = \frac{4}{3}\pi r^3 \quad , \text{But } r = r_1 + r_2 \text{ and thereof:}$$

$$P.F = \frac{4}{3}\pi(r_1 + r_2)^3 \qquad (11) \qquad \frac{Z}{V}$$

Where Z: the number of atoms per unit cell, which is equal to 2 in the rutile phase, 8 in the brookite phase, and 4 in the anatase phase.

r_1 and r_2: the radius of the titanium atoms, Ti and O, respectively.

Thus, the empty part of the titanium dioxide lattice appears according to Table (2).

We notice that in contrast to the increase in the cell size, the compaction coefficient decreased with the increase in the percentage of doping. It is clear that from the equation (10) that there is an inverse proportion between the cell size and the atomic packing fraction, while the latter is directly proportional to the size of the atoms in the compound, which differed according to the phase due to the difference in the number of atoms in the compound. Z unit cell between the three resulting phases. The highest value of the compaction coefficient was for the rutile phase PF = 0.0596, whose value was constant for all doped samples, due to the stability of the cell size of the doped samples in the rutile phase, where the crystal lattice constants of the rutile phase maintained their values by changing the doping ratios. As for the sample taken in phases together, the

Table 1: The atomic packing fraction of pure and lead-doped TiO2 powders with ratios (x = 0.2 - 0.5 - 0.7 - 0.9 g) taken for each phase separately and for the phases together.

Packing fraction **PF**					
The Products	TiO2:Pb (x=0.2)	TiO2:Pb (x=0.2)	TiO2:Pb(x=0.5)	TiO2:Pb(x=0.7)	TiO2:Pb (x=0.9)
Rutile	0.0595	0.0596	0.0596	0.0596	0.0596
Anatase	0.055	0.055	0.055	0.055	0.054
Brookite	0.0564	0.0563	0.0563	0.0563	0.0558
All phases	0.057	0.057	0.057	0.057	0.0565

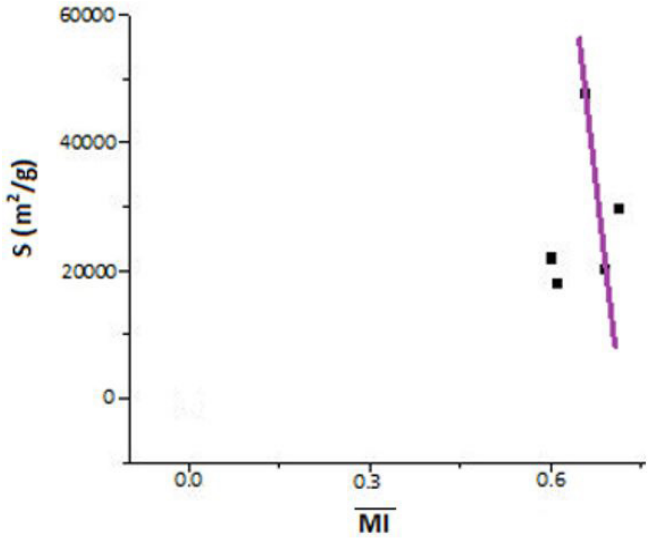

Figure 6: Specific surface area (m²/g) S of pure and lead-doped titanium dioxide samples with different ratios (x = 0.2 – 0.5 – 0.7 – 0.9) in terms of mean morphology index (MI)¯taken together (anatase, brookite and rutile). We note from Figures 4 and 5 that the average morphology index is directly proportional to the average crystal size of the phases together D¯ which falls within the range [57.379-85.370 nm] and inversely proportional to the specific surface area S which falls within the range [47842.78 - 18191.06 m²/g] In this order, this agrees with the researcher [19]. The increase in the morphology index is due to the increase in the grain size due to the effect of lead impregnation, which caused the increase in the denominator of the relationship [13], which expresses the morphology index.

Table 4: Tabular figures giving the values of the morphology index for pure and lead-doped TiO2 powders with the proportions (x = 0.2- 0.5 - 0.7 - 0.9 g).

Pure TiO_2		TiO_2:Pb (x=0.2)		TiO_2:Pb (x=0.5)		TiO_2:Pb (x=0.7)		TiO_2:Pb (x=0.9)	
2θ (deg)	MI (unit less)	2θ (deg)	MI (unit less)	2θ (deg)	MI (unit less)	2θ (deg)	MI (unit less)	2θ (deg)	MI (unit less)
32.100	0.500	32.1	0.485	32.1	0.572	32.1	0.572	32.1	0.445
42.355	0.530	33.6	0.931	33.6	0.572	33.6	0.400	33.6	0.400
46	0.572	36.7	0.800	36.7	0.889	36.7	0.572	35.7	0.667
48.50	0.500	37.4	0.667	37.4	0.667	37.4	0.500	36.7	0.728
51.80	0.667	42.36	0.572	42.355	0.667	42.355	0.572	37.4	0.800
64.4	0.616	46	0.690	46	0.667	46	0.500	39.6	0.667
67	0.500	48.5	0.445	48.5	0.572	48.5	0.625	42.355	0.667
71	0.616	51.8	0.800	51.8	0.800	51.8	0.541	46	0.667
73.7	0.800	57.7	0.667	52.3	0.800	57.7	0.667	46.5	0.800
74.9	0.667	64.4	0.572	57.7	0.500	62.3	0.667	48.5	0.607
76.2	0.572	67	0.625	64.4	0.667	64.4	0.656	52.3	0.910
83	0.572	71	0.690	67	0.800	66.4	0.800	53.5	0.667
83.7	0.800	71.9	0.625	71	0.728	67	0.500	54.5	0.656
$\overline{MI}$ For all phases	0.609	72.4	0.770	72.4	0.834	70	0.800	57.7	0.667
		73.7	0.690	73.7	0.770	71	0.500	61.215	0.785
		74.9	0.690	74.9	0.889	71.9	0.770	61.250	0.834
		76.2	0.646	76.2	0.667	72.4	0.770	64.4	0.800
		83	0.598	83	0.667	73.7	0.667	66.4	0.667
		83.7	0.770	83.7	0.728	74.9	0.572	67	0.889
		$\overline{MI}$ For all phases	0.656	$\overline{MI}$ For all phases	0.6892	76.2	0.667	70	0.834
						79.5	0.667	71	0.667
						83	0.500	71.9	0.800
						83.7	0.800	72.4	0.800
						$\overline{MI}$ For all phases	0.6004	74.9	0.667
								75	0.667
								78.4	0.834
								79.5	0.667
								83	0.572
								83.7	0.666
								87.6	0.800
								87.8	0.889
								88.9	0.667
								$\overline{MI}$ For all phases	0.712

Table 2: The blank fraction ratio of pure and lead-doped TiO2 powders with ratios (x = 0.2 - 0.5 - 0.7 - 0.9) taken for each phase separately and for the phases together.

Packing fraction **PF**					
The Products	TiO2:Pb (x=0.2)	TiO2:Pb (x=0.2)	TiO2:Pb(x=0.5)	TiO2:Pb(x=0.7)	TiO2:Pb (x=0.9)
Rutile	0.9405	0.9404	0.9404	0.9404	0.9404
Anatase	0.945	0.945	0.945	0.945	0.946
Brookite	0.9436	0.9437	0.9437	0.9437	0.9442
All phases	0.943	0.943	0.943	0.943	0.9435

Table 3: Specific surface area of pure and lead-doped TiO2 powders with ratios (x = 0.2- 0.5 - 0.7 - 0.9).

S (m2/g)					
The Products	TiO2:Pb (x=0.2)	TiO2:Pb (x=0.2)	TiO2:Pb (x=0.5)	TiO2:Pb (x=0.7)	TiO2:Pb (x=0.9)
Rutile	39803.97	16794.1	28846.96	42814.7	14064.61
Anatase	12948.71	18513.63	17333.94	71061.38	24710.2
Brookite	36586.95	30862.54	14834.62	29652.25	15798.35
All phases	29779.88	22056.76	20338.51	47842.78	18191.06

largest value of the packing fraction was for the sample with the highest doping ratio 0.9g, but the cell size was the highest value and also the theoretical density had the lowest value at that value, which amounted to 4.069 g / cm³ among the samples and The reason for this is only the significant decrease in density for the two phases of anatase and brookite, which was greater for the stage of brookite in particular, and this corresponds to the increase in cell size for each of the previous two phases, knowing that the phase of brookite is the most present in the recombinant compound in this research.

$$SSA = \frac{SA_{part}}{V_{part} * density} \quad (11)$$

$$S = \frac{6*10^3}{D\ \rho\ X-ray} \quad (12)$$

S: the specific surface area is m²/g.

V_{part}: particle size in nm3.

SA_{part}: surface area in nm2.

D: crystal size unit nm.

$\rho_{(X\text{-ray})}$: The theoretical density of the X-ray diffraction spectrum for powders is g/cm3.

Since the morphology index MI is related to both the particle size D and the specific surface area S, we calculated the MI for pure and lead-doped titanium dioxide powders using the equation (12) [19]:

$$M.I = \frac{FWHM_h}{FWHM_h + FWHM_p} \qquad (13)$$

M.I: morphology index.

$FWHM_h$: The highest value of the full width at half the intensity (FWHM) measured in radians (rad). FWHMp: Full width values at half maximum intensity (FWHM) measured in radians (rad).

The range of MI for pure TiO2 powder ranges within the range [0.500-0.800]. As for TiO2 powder doped with lead, it belongs to the range [0.400-0.931], we notice that the range expands between the lowest and largest index values compared to the pure sample, and the rutile phase has the highest value of the morphology index. MI = 0.931 at 0.2 lead impurity, followed by the value that we obtained in the sample impregnated with lead by 0.9 to reach MI = 0.910. With an increase in lead impurity, there was a clear decrease in the width of the peaks, which was followed by an increase in the morphology index.

Figure 6: Specific surface area (m2/g) S of pure and lead-doped titanium dioxide samples with different ratios (x = 0.2 – 0.5 – 0.7 – 0.9) in terms of mean morphology index (MI) taken together (anatase, brookite and rutile). We note from Figures 4 and 5 that the average morphology index is directly proportional to the average crystal size of the phases together $\bar{D}$ which falls within the range [57.379-85.370 nm] and inversely proportional to the specific surface area S which falls within the range [47842.78 - 18191.06 m2/g] In this order, this agrees with the researcher [19]. The increase in the morphology index is due to the increase in the grain size due to the effect of lead impregnation, which caused the increase in the denominator of the relationship [13], which expresses the morphology index.

Conclusion

In this research, we completed the study of some physical properties of titanium dioxide compound doped with lead in different proportions, after studying the X-ray diffraction (XRD) patterns of anatase and rutile with a quadruple crystal system and brookite with a rhombic crystal system based in the compound in order to know the changes that occur in this compound and to increase Investing it in new and wide fields and applications, we have studied the atomic compaction coefficient, morphology index and specific surface area for each phase of the pure titanium dioxide compound doped with lead in different proportions, and we also took measurements for the phases together. The morphology index, which falls within the range [0.500-0.800], confirmed that the compound belongs to pure titanium dioxide, and the morphology index for the doped samples fell within the range [0.400-0.931], and its highest value belonged to the rutile phase at the ratio of 0.2g, as it occurred. The specific surface area of the compound is within the range [47842.78 - 18191.06 m²/g], and the anatase phase had the highest value of the specific surface area compared to the other two phases, which amounted to S = 71061.38 m²/g. As for the atomic compaction ratio taken for the phases together, it fell within the range [0.0565- 0.0570]. The results were identical with many researchers.

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