



## Literature Study of The Effect of Aluminum Doped ZnO Nanoparticles on Structure and Gap Energy

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**Abstract,** *Nanomaterials have a variety of uniqueness and attractiveness, it is considered necessary to carry out literature study, beside the laboratory investigation, among others, on the nanoparticle structure and energy gap of ZnO nanoparticles doped with aluminum (Al) resulting from synthesis using various methods. In this study, the structure of the nanoparticles was divided into three types, namely crystal structure, crystallite size, and morphology. The crystal structure of the Al doped ZnO nanoparticles is hexagonal wurtzite. It is found, that, a decrease in crystallite size occurs as the doping of Al into ZnO increases to its solubility limit. A similar pattern also occurs in particle size. The high concentration of Al doping results in agglomeration. The morphology of Al doped ZnO nanoparticles in some references of this study are rod, spherical, and irregular spheres. The energy gap value of Al doped ZnO nanoparticles increases with the increasing concentration of Al doping in ZnO. So, it can be said, that Al as a doping material has an influence on the structure and optical properties of ZnO nanoparticles.*

**Keyword:** gap energy; nanoparticle; structure; ZnO

### 1. INTRODUCTION

Over time, technology evolves into increasingly complex [1]. The technology that emerged is nanotechnology [2]. One of the important sub-fields in nanotechnology that has a relationship with energy is nanofabrication which basically contains the process of storing and transferring energy [3]. The level of reactivity and efficiency of nanoparticles will generally increase as they decrease in size [4].

Technology development by utilizing optical principles posed by nanoparticles one of which is photoanoda in DSSC (Dye Sensitized Solar Cell) [5]. DSSC is one of the developments of solar cell technology. Semiconductor materials used mostly are TiO<sub>2</sub> [6]. TiO<sub>2</sub> has a gap energy value range of 3.20 eV – 3.51 eV [5]. However, if exposed to the human body, TiO<sub>2</sub> has a less good effect as it can be the cause of tumors and cancer [7], so it needs a replacement material that can cover this deficiency. One of the materials that is widely developed to be reviewed from the size of the nanometer is ZnO (Zinc Oxide) / Zinc Oxide. ZnO as a semiconductor material is widely applied to optical technology which has several advantages that are non-toxic, if exposed to the body and only require a low cost to process it [8] [9]. Thus, it can be said, that ZnO can be an alternative to TiO<sub>2</sub> in the context for DSSC photoanoda with ZnO gap energy value at nanometer size is 3.26 eV. It takes a doping to

increase the energy value of the gap for DSSC applications [10]. When ZnO doping with Al, the energy gap becomes greater than the pure ZnO [11]. This indicates that ZnO is suitable as a replacement for TiO<sub>2</sub> in terms of gap energy value for DSSC [12].

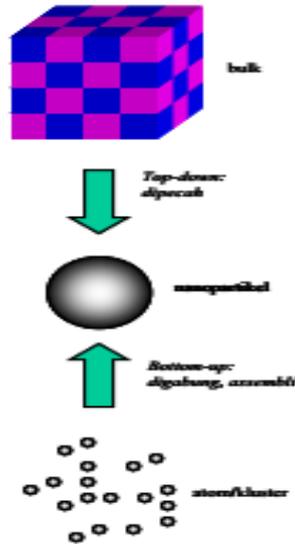
Given that there has been a lot of research on the influence of aluminum doping on ZnO nanoparticles on crystal structure and gap energy, a literature study that summarizes the results is needed to compare and analyze the results of research that in his experiments used different methods so that a general and complete picture of the influence of aluminum doping on ZnO nanoparticles is needed.

### **Nanotechnology and Nanoparticles**

The term "nano" originated in ancient Greek. Literally, this mention means "very small". The dimensions of the nano are below the macroscopic range of 1 nanometer (nm) to 100 nm. The application of science is done by reviewing a small thing called nanotechnology. [13]. In the development of nanotechnology, the most popular main thing in it is nanoparticles. Nanoparticles are particles that have a size on a nanometer scale [14]. Quantum dot is one example that represents the quantum effect event of a particle that is several nanometers in size. In this quantum dot, electron locking events occur in quantum dots which then produce a quantized spectrum of energy. Nanoparticles in general can be classified by dimension, morphology, composition, uniformity, and agglomeration [4].

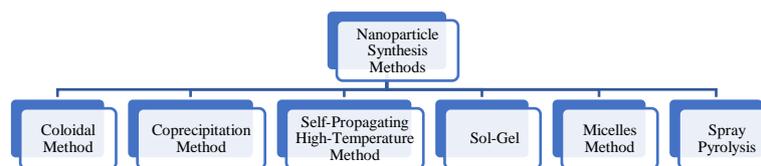
### **Synthesis of Nanoparticles**

There are a variety of techniques that can be performed to create a nanometer-sized particle. In principle, the manufacture of nanoparticles is divided into two namely Bottom-up and Top-down (Figure 1). Bottom-up can also be called a constructive method which is a method that builds material from atomic scale to nanoscale cluster (nanoparticle). Then, for the category of top-down can also be called destructive method, which is a method that reduces or breaks chunks of material (bulk) into particles with nano scale. Several synthesis methods can be performed to create nanoparticles (Figure 2).



**Figure 1 Nanoparticle synthesis process**

Source: [15]

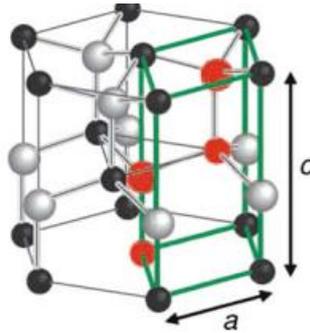


**Figure 2 Nanoparticle synthesis methods**

Source: [16]

## Zinc Oxide

Zinc Oxide (ZnO) is one of the versatile materials with technological potential as diverse as transparent electrode in solar cells, thin layer of transparent transistors, photonic crystals with adjustable tape gaps, and anti-reflection coatings. There are several interesting properties that ZnO has, namely high chemical stability, non-toxic, and biocompatible. ZnO is a II-VI semiconductor with direct band gap type. The amount of connective energy that ZnO has at room temperature is 60 meV. The ZnO is an N-type semiconductor with maximum optical transparency within the visible signal spectrum region. ZnO has a wurtzite hexagonal crystal structure with grid parameters  $a = 0,324$  nm and  $c = 0,5207$  nm [17]. Zn has an atomic radius of  $1,42 \text{ \AA}$  [18], while in cation form ( $\text{Zn}^{2+}$ ), it has an ion radius of  $0,74 \text{ \AA}$  [19]. For oxygen (O) it has an atomic radius of  $0,48 \text{ \AA}$  [18] and in anion form ( $\text{O}^{2-}$ ), it has a radius of  $1,40 \text{ \AA}$  [19]. The ideal ratio for two lattice parameters possessed by a wurtzite structured crystal is 1,633 [20].



**Figure 3 ZnO hexagonal wurtzite lattice parameter**

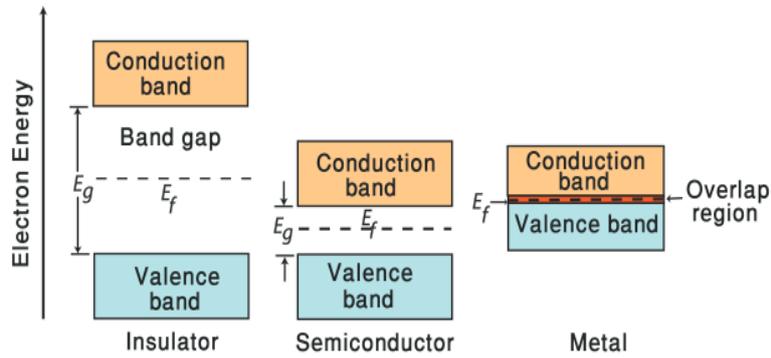
Source: [20]

### **Aluminum**

Aluminum (Al) is one of the elements of the type of metal. This element is non-toxic, so it is safe for humans to be applied into a thing. In addition, aluminum is also a metal that is still within the safe threshold if in contact with for the environment [12]. It has a radius of 1.18 Å [18]. In the form of  $\text{Al}^{3+}$  ions, it has a radius of 0,50 Å [19]. Aluminum has a Face Centered Cubic (FCC) crystal structure. Aluminum structures can be transformed (allotropic) at 870 °K and 350 °K. The thermal and mechanical properties possessed by aluminum according to ASM Handbook Volume 2 (1990) in [21] have melting points at 660,4 °C and boiling points at 2494 °C. Then, thermal conductivity at 25 °C and 660,4 °C, respectively, namely 247 W/m.K and 90 W/m.K. Pure aluminum can be obtained from alumina compounds ( $\text{Al}_2\text{O}_3$ ) by electrolysis of its salts that have been diffused with its metastable structure. Aluminum also has a modulus elasticity value of 2386,67 N/mm<sup>2</sup>, density 2,7 g/cm<sup>3</sup>, and has a hardness of 245 HB [22].

### **Semiconductor Gap Energy**

Band gap is an energy range in which no electronic activity occurs. Band gap or commonly referred to as gap energy can be used to distinguish the type of material based on its electrical conductivity, among others insulators, conductors, and semiconductors. In insulator materials, there is a large gap between the valence band and the conduction band. However, the opposite situation occurs in the conductor material, namely the valence band with the conduction band narrowing each other. In semiconductor materials, there is a small gap between the valence band and the conduction band (Figure 4) [23].

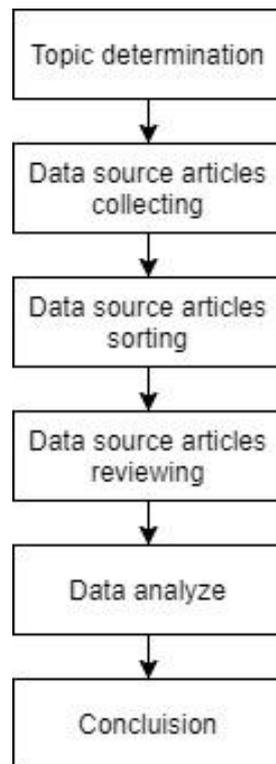


**Figure 4** Width of gap energy in each material

Source: [23]

## 2. METHOD OF RESEARCH

A series of phases that are used in this research with some step for analysis:



**Figure 5** General step of research

## 3. RESULT AND DISCUSSION

### Crystal Structure

**Table 1** Crystal structure of Al doped ZnO

Reference	Synthesis Method	Database Reference	Crystal Structure
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[24]	Sol-Gel Spin Coating	JCPDS 89-7102	Hexagonal Wurtzite
[25]	Sol-Gel	JCPDS 36-1451	Hexagonal Wurtzite
[26]	Sol-Gel	JCPDS 80-0075	Hexagonal Wurtzite
[27]	Sol-Gel	JCPDS 36-1451	Hexagonal Wurtzite
[28]	Sol-Gel	JCPDS 36-1451	Hexagonal Wurtzite
[29]	Coprecipitation	JCPDS 36-1451	Hexagonal Wurtzite
[30]	Hydrothermal	JCPDS 01-070-2551	Hexagonal Wurtzite

Source: Author's data

**Table 2 Lattice parameter of Al doped ZnO**

Referenc e	Doping Al (%)	a (Å)	c (Å)	c/a	Crystal Structure
[30]	0	3,25 1	5,208	1,602	Hexagonal Wurtzite
	0,5	3,24 9	5,204	1,602	Hexagonal Wurtzite
	1	3,25 1	5,208	1,602	Hexagonal Wurtzite
	2	3,25 0	5,207	1,602	Hexagonal Wurtzite
[28]	0	3,26	5,20	1,595	Hexagonal Wurtzite
	2	3,20	5,17	1,616	Hexagonal Wurtzite
	6	3,17	5,12	1,615	Hexagonal Wurtzite

Source: Author's data

Determination of the type of nanoparticle structure of Al doped ZnO in various reference articles is done by means of testing samples using XRD which then produces a graph between  $2\theta$  ( $^\circ$ ) with intensity (a.u) which can also be called a diffractogram [31]. In table 1, the database referred to by researchers in some reference articles is JCPDS. Thus, with the database, miller's

index in each sample in each literature can be used as material to know that the crystalline structure of Al doped ZnO nanoparticles is hexagonal wurtzite. Another indication to find out the type of crystal structure of the Al doped ZnO nanoparticles through its lattice parameter ratio ( $c/a$ ).

There is lattice parameter ratio data, which is listed in table 3.2, which can be used as supporting data to determine the crystal structure of Al doped ZnO. The ratio value of lattice parameters ( $c/a$ ) with a range of 1,593 to 1,633 is the ratio value possessed by the wurtzite crystal structure to ZnO nanoparticles [32] [20]. So, according to the data in table 2, nanoparticles Al doped ZnO in [30] and [28] are hexagonal wurtzite. And it can be said, that the crystal structure in the reference article did not change the structure, although given al doping with a different percentage. Then, in the reference article for the data on determining the crystal structure of ZnO nanoparticles of Al doping as stated in table 3. 1 other than [30] and [28], have no information on the ratio of grid parameters ( $c/a$ ). However, the crystalline structure of the Al doped ZnO nanoparticles can still be known through the similarity of miller index values listed on the XRD diffractogram between one article and another.

The ratio value of the grid parameter ( $c/a$ ) with a range of 1,593 to 1,633 is the ratio value possessed by the wurtzite crystal structure for ZnO nanoparticles [32][20]. So, according to the data in table 3.2, nanoparticles ZnO doping Al in [30] and [28] is hexagonal wurtzite. And it can be said, that the crystal structure in the reference article did not change the structure, although given al doping with a different percentage.

### Crystal Structure

**Table 3 Crystallite size of Al doped ZnO**

Reference	Synthesis Method	Doping Al (%)	Crystallite Size (nm)
[24]	Sol-Gel Spin Coating	0	38
		1	27
		2	18
		3	12
		4	9
		5	7
[25]	Sol-Gel	0	78,7
		3	49,6
		6	29,9

		9	35
		12	52,4
[26]	Sol-Gel	0	28,65
		2	20,25
		3	20,22
		4	10,78
[27]	Sol-Gel	0	32
		0,5	27,2
		1	24,1
		2	14,9
[28]	Sol-Gel Dip Coating	0	19,9
		2	15,2
		6	9
[29]	Coprecipitation	0	47
		0,5	43
		1	42
		1,5	39
		2	40
		2,5	46
[30]	Hydrothermal	0	194 ± 4
		0,5	15 ± 1
		1	35 ± 1
		2	47 ± 1

Source: Author's data

This decreased crystallite size event as in table 3 that occurs in each addition of Al doping percentage into ZnO can occur because the radius of the ion of Al<sup>3+</sup> (0,5 Å) is smaller than the ion radius of Zn<sup>2+</sup> (0,74 Å). From this condition, if the two to be as one, there will be a substitution of ions between the two. Thus, with Al doping will result in the narrowing of the lattice. However, there are different conditions, namely an increase in crystallite size at the Al 9% and 12% doping percentage [25]. This is due to the threshold of the solubility of Al atoms in the ZnO crystal structure. As a result, the Al<sup>3+</sup> ion that became doping for ZnO is no longer the cause of the atomic substitution event against Zn<sup>2+</sup> ion in the hexagonal wurtzite crystal

structure, but the Al atom will create an atomic interstition event. The large number of interstitial doping atoms will lead to the appearance of crystal distortion and decreased mobility of Al as a doping material. Therefore, excessive Al doping will be the cause of the increasing size of crystallites [33].

A similar condition occurs in the size of crystallites listed in [29] by the synthesis of coprecipitation methods. The size of crystallites in Al doped ZnO was 1,5%. Al doping value below 1,5% (0 – 1,5%) into ZnO shows a pattern of crystalline size values that decrease as al doping values increase into ZnO. But the data showed the opposite pattern for Al's doping scores above 1,5% (2 and 2,5%). The event of increasing the size of crystallite in this article, which starts from the percentage of Al doping by 1,5% to 2,5% is due to the limit of al solubility level in ZnO crystals. Crystallite size events also occurred in [30] where crystallite size increased as the percentage of Al doping increased (0,5; 1; and 2%) [33].

### Morphology

*Table 4 Morphology of Al doped ZnO*

Reference	Synthesis Method	Doping Al (%)	Morphology	$\bar{D}$ (nm)
[25]	Sol-Gel	0	Nanorod	525,218
		3	Irregular sphere	480,297
		6	Irregular sphere	312,696
		9	Irregular sphere	550,810
		12	Irregular sphere	197,121
[27]	Sol-Gel	0	Irregular sphere	291,843
		2	Irregular sphere	426,520
[28]	Sol-Gel Dip Coating	0	Spherical	10,736
		2	Spherical	8,424
		6	Spherical	6,556
[34]	Dip Coating	0	Spherical	189,571
		1	Spherical	153,540
		3	Spherical	149,268
		6	Spherical	130,654

Source: Author's data

Morphology that indicates the agglomeration or clumping of a ZnO nanoparticle of Al doping as in [25] and [27] can happen because of several things. The first reason is that it can happen because it is already at the limits of solubility of Al as a doping material from ZnO. Low solubility will cause particle movement to be worse. Because, it is known that particles that have a smaller size will have a larger surface energy, so that the particles will easily clump. Al's low solubility occurs due to the increasing percentage of Al doping in ZnO nanoparticles as does the case with crystallite size. As a result, particles will form agglomerations with diverse models such as simply forming colonies or forming unity into large particles. Then, another factor that can be the cause of agglomeration events, namely the presence of impurities. This impurity can be derived from precursor substances that become the raw material of synthesis. The abundance of impurity substances will add many factors that will potentially integrate in terms of increasing crystal growth. The size of the crystals formed will determine the size of the formed particles [35] [36].

When reviewed based on the morphological form of the Al doped ZnO nanoparticles in various reference articles as listed in table 4, there are two main underlying forms of rod and sphere. The nanorod form is present in [25] in the state of ZnO nanoparticles without Al doping. Nanorods are formed as a result of the Ostwald Ripening mechanism. This mechanism occurs because crystals that have a small size tend to dissolve which then attach to a larger surface. Then, larger crystals will grow over time as smaller crystals are no longer present due to previous processes. Thus, small crystals will form large particles in this case are nanorods [37].

Then, after being granted Al doping, the basic form of the nanoparticles changed to sphere. This change in shape can occur due to differences in conditions for solvents in dissolving precursors i.e.the presence and absence of Al as a doping material [36]. Solvents that in fact have a certain polar level will stick to the precursor crystals in a particular crystal field which will inhibit the growth of c-side crystals. These obstacles result in nanoparticles having sphere shape [25].

## Gap Energy

**Table 5 Gap energy of Al doped ZnO**

Referenc e	Synthesis Method	Doping Al (%)	Gap energy (eV)
[38]	Sol-Gel	0	3,47
		0,5	3,43
		1	3,44

		2	3,69
		4	3,64
[34]	Dip Coating	0	3,55
		1	3,68
		3	3,82
		5	3,25
[39]	Atomic Layer Deposition (ALD)	0	3,271
		0,97	3,302
		2,06	3,399
		2,26	3,434
		2,5	3,458
		2,81	3,476
		3,2	3,505
		3,71	3,531
[27]	Sol-Gel	0	3,28
		0,5	3,37
		1	3,4
		2	3,44
[30]	Hydrothermal	0	3,28
		0,5	3,37
		1	3,40
		2	3,44

Source: Author's data

The value of gap energy as one of the optical properties that can be reviewed by Al doped ZnO nanoparticles based on table 5 has an increased value pattern in line with the increasing percentage of Al doping in ZnO nanoparticles. This can happen because Al if used as a doping material for ZnO, he acts as an n-type doping material [40]. If a material is no exception ZnO if given doping in the form of n-type material, then the value of energy gap will increase in line with the increasing value of doping as seen in the table 5. It is like the research of [41] that the increase in the energy gap value of ZnO nanoparticles coincides with an increase in the percentage of Al doping into which events like this are described as the Burstein-Moss effect. The Burstein-Moss effect is a condition when the  $Al^{3+}$  ion as a doping agent has a role as a

Zn<sup>2+</sup> ion substitute and as an interstition atom will be the cause of increased gap energy due to the increased concentration of carriers due to Al as an electron donor element. Al doping as an n-type doping material will have an effect on the lowest energy level of the conduction band, so that it will increase the gap value between the valence band and the conduction band, and fermi energy will be above the base of the conduction band [42] [43]. Thus, based on this explanation, it is proven that along with the increasing percentage of Al doping in ZnO nanoparticles will result in the energy gap it has will increase.

The increased energy gap has been linked to a decrease in the size of ZnO nanoparticle crystallites with an increased percentage of Al doping. This is due to the common effect called quantum confinement effect. This effect can connect the size of crystallite with the energy value gap because the relationship between the two is inversely proportional. If there is a substance that is getting smaller in the nanoscale, then the gap energy of the substance will increase [44].

A slightly different thing about the energy value gap occurred in [34] due to a decrease in the value of gap energy at the al 5% doping percentage into the ZnO nanoparticles in the table 3.5 from 3,82 eV to 3,25 eV. According to [45], the decrease in the energy gap can occur due to the percentage of doping is considered an excessive amount of doping. It is indicated that it can be the cause of the increase of impurity that will react with the nanoparticles Al doped ZnO which then forms a new state as a impurity band that appears under the conduction band, so that impurities substance will contribute to the narrowing of the band gap [41].

In non-doping conditions, ZnO nanoparticles have a gap energy value of 3,26 eV. Then with Al doping, the energy gap of the ZnO nanoparticles increases to 3,82 eV (table.5). This is evidence that the Al doped ZnO nanoparticles have one of the appropriate criteria for replacing TiO<sub>2</sub> as a DSSC material in terms of its gap energy value.

#### **4. CONCLUSION**

Based on the results of the literature studies that have been done, it can be concluded that Al doping on nanoparticles has no effect on the crystalline structure of ZnO nanoparticles based on the ratio of crystal lattice parameters (c/a) and miller index, i.e remains hexagonal wurtzite for all variations in al doping percentage. Influence on crystalline size, i.e along with increasing the percentage of doping Al into ZnO nanoparticles to a certain limit of solubility, crystalline size value decreases due to Al<sup>3+</sup> ion substitute on Zn<sup>2+</sup>. The size of crystallite increases when Al crosses the atomic solubility boundary due to the intercession of Al atoms in the lattice between

Zn and O atoms. The resulting nanoparticle shape is spherical, irregular sphere, and rod, Rod shape occurs due to the Ostwald Ripening mechanism. Spherical and irregular sphere forms occur because the unidirectional growth of c lattice parameters is inhibited. Particle size decreases along with the increase in the percentage of Al-doping. Decrease in crystalline size and particle size along with increasing percentage of Al doping occurs because particles are composed of many crystallites. Nanoparticle agglomeration occurs due to decreased levels of nanoparticle solubility due to the percentage of doping that exceeds the limits of solubility. The energy gap value of the ZnO doping Al nanoparticles increased along with the increasing percentage of Al doping as an n-type element due to the Burstein-Moss effect. Al doping ZnO nanoparticles meet criteria as TiO<sub>2</sub> substitutes for DSSC materials based on their gap energy value

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