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# FACTORS AFFECTING THE SIZE OF $AL_2O_3$ NANOPARTICLES SYNTHESIZED BY SOL-GEL PROCESS

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## ABSTRACT

Aluminum oxide, $Al_2O_3$, nanoparticles were synthesized from alcoholic solution of Aluminum Chloride, $AlCl_3$, and ammonia via sol-gel process. Molar ratio of $NH_3$: $AlCl_3$ has been changed to control the size of the resultant nanoparticles. The structures of $Al_2O_3$ nanoparticles were investigated and confirmed using Fourier Transform Infrared spectroscopy (FT-IR) and X-ray diffraction (XRD), and their morphology was studied via high resolution transmission electron microscopy (HR-TEM). The HR-TEM images showed that the $Al_2O_3$ nanoparticles have spherical shape with a particle size ranging from 7 nm to 50 nm depending on the precursor's concentrations. It is found that higher ammonia and water concentration resulted in increasing the particle size of the $Al_2O_3$ nanoparticles. Micro particles of $Al_2O_3$ were obtained when the way of the precursors' addition has been altered; adding the ammonia to the alcoholic solution of the $AlCl_3$. The proposed mechanism behind the nanoparticles preparation as well as the dependence of their size on the selected factors has been discussed.

KEYWORDS: Sol-Gel process, $Al_2O_3$ Nanoparticles, Size, Factors, XRD, TEM.

## 1. INTRODUCTION

The great interests in nano science and nanotechnology lie in the discovery and characterization of novel materials with at least one dimension in nanometer scale (approximately 1–100 nm in length) [1]. At nano scale dimensions, the properties of these materials may differ significantly compared to their bulk counterparts. The tiny particle size, and the particle boundary and crystalline state of nano materials lead to increase the surface area of these materials and consequently enhance their reactivity. Furthermore, nano materials have attracted much attention due to their unique

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electronic, optical, magnetic and mechanical properties. These fascinating properties employed these types of materials in different application and in various disciplines ranging from engineering materials to biotechnology and energy storages [2, 3].

The unique properties and the potential applications of nano systems have forced the materials scientists and engineers to make significant developments in their synthetic routes [4-6]. Thus, different techniques with different starting materials are reported in the preparation of nanoparticles such as sol-gel technique [7, 8], chemical precipitation technique [9, 10], electro deposition technique [11, 12], and thermal decomposition [13]. These different approaches have resulted in production of variety of particles differing in their elemental composition, shape, size, and chemical or physical properties. In general, the nanoparticles preparation methods are classified into two main categories; physical and chemical methods. The first approach is generally based on the principle of sub-dividing bulk precursor materials into smaller nanoparticles, and it belongs to a so called top-to-bottom approach. However, the chemical approach, one of bottom-to-top approach, involves the preparation of metal oxides or composite metal oxides by the chemical reaction. It has proven that synthesis of nanoparticles by chemical methods is more effective and produces mono-dispersed particles compared to physical methods.

One of the most common chemical methods is the sol-gel method in which the metal oxides are obtained from the hydrolysis of the metal alkoxide or the metal salts in the presence of acid or base as catalysts [14]. Sol-gel is one of the most successful techniques to fabricate different nanoparticles with controlled shape and porosity. The advantages of that method lie in the fact of putting the homogeneous mixing of different ingredients into practice. Moreover, other advantages such as being a versatile process [15] with high purity, good homogeneity, and low processing temperature can be taken into account for this synthetic technique [16]. However, its drawbacks include the high cost of some metal alkoxides; the primary precursors used in such preparation methods, and the complication of the treatment procedure for the samples which increases the complexity of preparing process.

The nano material chosen in this study was Aluminum Oxide ($Al_2O_3$). Its high hardness, high melting temperature, excellent electrical insulation and good thermal properties make it the chosen material for a wide range of applications such as manufacturing of transparent ceramics, packaging materials, cutting tools, plastic, tape, grinding belt; paint, rubber, plastic wear-resistant reinforcement, etc [17, 18]. It was used in a previous study to enhance the flow boiling heat transfer coefficient of R134a refrigerant in vapor compression refrigeration cycle through the preparation of $Al_2O_3$/Polyol Ester oil (POE) nanofluid [19]. The particles of $Al_2O_3$ can be synthesized by several techniques such as sol-gel, ball milling, pyrolysis, sputtering, hydrothermal, and laser ablation [20-24]. Their synthesis by sol-gel method involves using different precursors: aluminum triisopropylate in a hydrolysis system consisting of octanol and acetonitrile [25], aluminum nitrate – in aqueous medium [26, 27], aluminum secondary butoxide - in an alcoholic medium [28].

The main scientific objective of the current research is to study the factors affecting the particle size of $Al_2O_3$ nanoparticle synthesized based on the sol-gel process. The factors include varying the initial concentration of the reactants (ammonia and water) and altering the sequence of their addition. The prepared nanoparticles will be characterized using different techniques to validate their synthesis. The proposed mechanism that elucidates the particle size dependence on the suggested factors will be also discussed here.

## 2. EXPERIMENTAL METHODS

### 2.1 Materials

The materials used in the present study are; aluminum trichloride, $AlCl_3$, (95% purity, Fluka), ammonia solution (33%, Fluka), absolute ethanol (99.5% purity, Adwic), and deionized water. The starting materials and solvents were used as received and without further purification.

## 2.2 Synthesis of $Al_2O_3$ Nanoparticles

$Al_2O_3$ nanoparticles were prepared via sol-gel technique using 0.1 M ethanolic solution of $AlCl_3$, ammonia solution (33%) as a base catalysis, and ethanol as an alcoholic medium. The synthetics route involves the addition of ethanolic solution of $AlCl_3$ drop by drop to a reaction vessel containing an appropriate amount of ethanol, deionized water and ammonia. The mixture was stirring at room temperature using magnetic stirrer. After few minutes, a gelatinous material of aluminum hydroxide, $Al(OH)_3$, has been obtained. This gel was kept under vigorous stirring to maturate for 24 hours. After the completion of stirring time, the gel formed was centrifuged using a centrifuge unit (Hitachi Himac CR 22G) and washed several times by absolute ethanol. The collected samples were dried in an oven at 100 $^oC$ for 10 hours, and then they were calcined in a burning furnace at 1000 $^oC$ for 4 hours. The calcined solid samples were crushed smoothly to get fine homogeneous white powder. In the current work, five different samples were prepared using different initial concentrations of the reactants as summarized in table 1. The first four samples, labeled $AO_{1-4}$, have been prepared by the addition of the alcoholic solution of $AlCl_3$ to the ammonia solution (as described above). However, the fifth sample, labeled $AO_{rev}$, has been prepared by altering the way of addition; the ammonia solution has been added gradually to the alcoholic solution of $AlCl_3$ containing suitable amount of water and ethanol as shown in Table 1.

Table 1. Sample codes and concentrations of the starting materials used in $Al_2O_3$ nanoparticles preparation with the range of the particle size obtained using the corresponding mixture.

| Sample code | $AlCl_3$ | $NH_3$ | $H_2O$ | Range of particle size (nm) |
|---|---|---|---|---|
| **$AO_1$** | 0.1 | 0.3 | 0.6 | 7-8 |
| **$AO_2$** | 0.1 | 0.3 | 1.2 | 10-15 |
| **$AO_3$** | 0.1 | 0.6 | 0.6 | 25-30 |
| **$AO_4$** | 0.1 | 0.6 | 1.2 | 50-60 |
| **$AO_{rev}$** | 0.1 | 0.3 | 0.6 | micro |

## 3. CHARACTERIZATION AND TECHNIQUES

The prepared nanoparticles have been characterized by several techniques to elucidate their structure and properties.

The synthesis of nanoparticles was confirmed by FT-IR spectroscopy. The infrared spectra of the prepared nanoparticles were recorded in the wavenumber range of 400–4000 $cm^{-1}$ using a Thermo Scientific spectrometer. Powdered samples being examined by FT-IR spectroscopy were prepared by grinding with potassium bromide (KBr) powder, and the mixture was then pressed into a disk.

Identification and characterization of samples based on their diffraction pattern was obtained via XRD. Thus, XRD spectrum of the prepared nanoparticles was collected by X-ray diffractometer (X'Pert PRO, PANalytical, Netherlands) using Cu Kα radiation in the angular region of $2\theta = 4^{o}$-$70^{o}$. The instrument was operated at 40KV and the spectra were recorded at scanning speed of $8^{o}$/min.

The morphology of samples structure and their average particle size was examined by HR-TEM (JEOL 2100, 200kV). To prepare samples for transmission electron microscopy (TEM), a drop of their colloidal suspension was placed on a copper grid coated with a carbon membrane. The solvent was evaporated at room temperature, leaving the nanoparticles on the grid.

## 4. RESULTS AND DISCUSSION

$Al_2O_3$ nanoparticles were prepared through hydrolysis and condensation of $AlCl_3$ through the sol-gel technique. This technique is widely used for preparation of metal oxide of controllable and uniform size for applications in materials science [29]. This may be due to its ability to control the hydrolysis and condensation reactions which in turn can affect the properties of the obtained nanoparticles. The following sections discuss the data obtained from the techniques used to characterize the obtained samples.

## 4.1 IR Spectra of $Al_2O_3$ Nanoparticles

Upon converting the $AlCl_3$ into $Al_2O_3$ powder, the former compound undergoes hydrolysis process in which its alcoholic solution reacts with water to form gelatinous aluminum hydroxide, $Al(OH)_3$, and hydrochloric acid, HCl, according to Eq. 1. After collecting the produced gel, the sample was dried at 100 $^oC$ then calcined at 1000 $^oC$. At this high temperature, the molecules get polymerized via condensation process, according to Eq. 2, forming white powder of $Al_2O_3$.

$$AlCl_3 + 3H_2O \rightarrow Al(OH)_3 + 3HCl \quad (1)$$

$$2Al(OH)_3 \rightarrow Al_2O_3 + 3H_2O \quad (2)$$

The obtained powder was characterized using FT-IR spectroscopy by which the infrared absorption spectrum of the prepared $Al_2O_3$ has been obtained over a wavenumber ranging from 400 to 4000 $cm^{-1}$ as represented in Fig 1. In this figure, it can be shown that there is a broad band at around 3400 $cm^{-1}$ which corresponds to the O-H absorption band due to the adsorption of water molecules on the particles surface. In addition, there is only one sharp peak at 570 $cm^{-1}$ which corresponds to the absorption of the stretching vibration of Al-O bond. There is no chance for appearing any other peak corresponds for organic impurities since the samples undergo heating at high temperature.

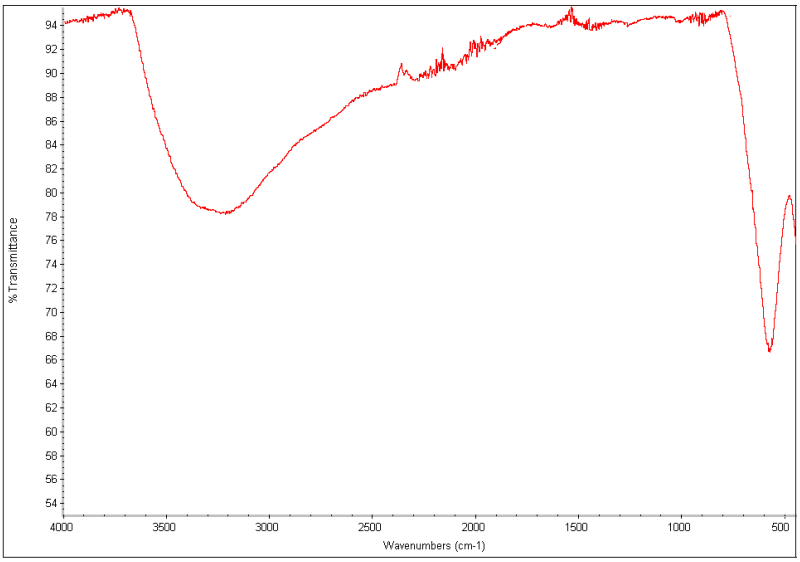



Fig. 1. FT-IR spectrum of the $Al_2O_3$ nanoparticles.

## 4.2 XRD Analysis of Nanoparticles

The crystalline structures of the powdered nanoparticles were studied and analyzed using X-ray diffractometer. A typical XRD plot is shown in Fig. 2. The diffraction angle and intensity of the sample's peak is well consistent with that of the previous reported XRD of $Al_2O_3$, and there are no peaks for impurities or any other precursor compounds were observed [30, 31]. The broad XRD pattern reveals the presence of γ-alumina [22, 25] as compared to the lines underneath the diffractogram of the sample.

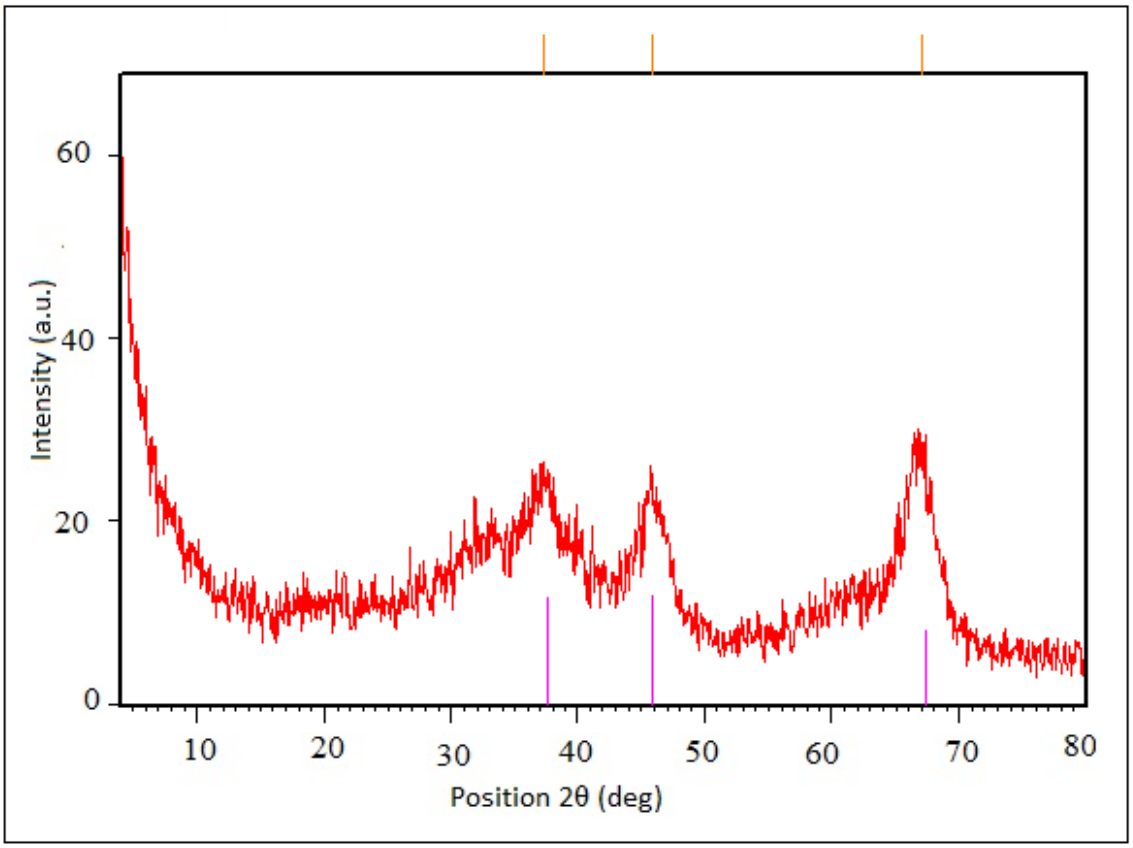
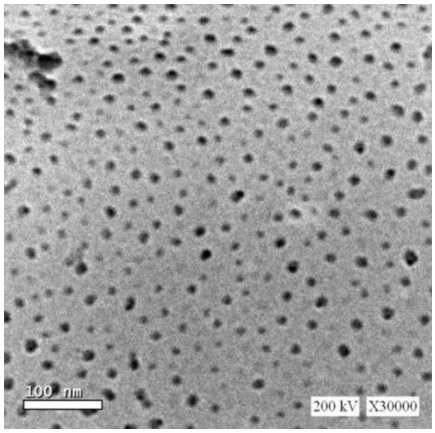



Fig. 2. XRD of the prepared $Al_2O_3$ nanoparticles.

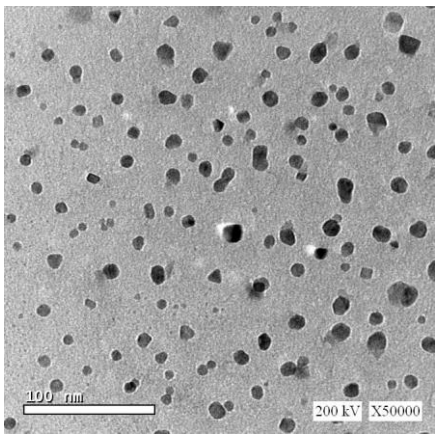
## 4.3 Morphologies of the Synthesized $Al_2O_3$ Nanoparticles Using TEM

The morphology of aluminum oxide was examined via TEM, and the obtained data was presented in Fig. 3. The TEM images confirmed that the $Al_2O_3$ particles were nearly spherical in shape and with an average particle size ranging from 7 nm to 60 nm

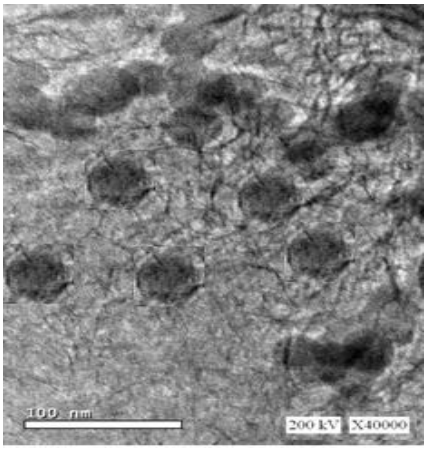
depending on the initial concentration of the reactants. The spherical shape of the nanoparticles is uniform at high concentration of ammonia, samples AO3 and AO4. Furthermore, the particle size of the sample labeled $AO_{rev}$ obtained from altering the sequence of reactants addition has large particle size which exceeds the range of TEM instrument. The reasons and mechanism behind all of the above observations will be discussed in the following section, section 5.



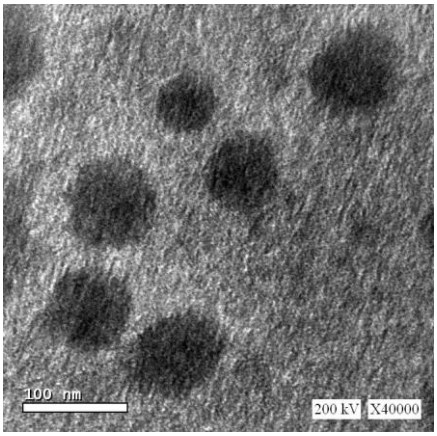

Sample $AO_1$: 7-8 nm




Sample $AO_2$: 10-15 nm




Sample $AO_3$: 35-40 nm




Sample $AO_4$: 50-60 nm

Fig. 3. TEM of $Al_2O_3$ nanoparticles with different average particle size.

## 5. MECHANISM OF NANOPARTICLES FORMATION AND FACTORS AFFECTING THEIR SIZE

Sol-gel process is a technique used to produce solid materials, especially metal oxides, from small molecules. In the preparation of $Al_2O_3$, it involves the hydrolysis of the $AlCl_3$ to form small particles (the sol), then the particles undergo condensation reaction via linking these small molecules into chains forming a network (gel). Generally, the rates of hydrolysis and condensation of sol-gel process depend on the pH of the reaction media, i.e. concentration of ammonia solution. In addition, the water concentration also affects these rates and consequently affects the properties and size of obtained particles. In general, changing the concentration of the precursors, temperature of the reaction, and pH of the reacting media are considered conditions determinant of nanoparticle growth [29].

Hydrolysis process is a slow reaction whether in an acidic or a basic medium. Using ammonia as a base catalyst will speed up the rate of hydrolysis and condensation of $AlCl_3$ resulting in faster kinetics and lager particle sizes [32]. At low pH, the rate of hydrolysis is faster than that of condensation, while under high basic conditions, the rate of condensation is faster than that of hydrolysis. Thus, by comparing samples AO1 with AO3, or samples AO2 with AO4, which have the same concentration of water and different ammonia concentrations, the solubility of the small particles at high ammonia concentration is higher than that at low concentration. This results in larger particle size of the obtained particles.

Herein, changing the water concentration has also affected the particle size of the obtained alumina. This can be noticed by comparing samples AO1 with AO2, or samples AO3 with AO4, which have the same concentration of ammonia and different water concentrations. It can be seen that the particle size increases as the water concentration increases. This can be attributed to two different reasons: increasing the water results in increasing the nucleation rate which in turn resulted in formation of numerous tiny sub-particles that can aggregate together by the action of H-bonding force forming larger particle size. The other reason is the increase in solubility of the

small particles at high water concentration which results in particles with large particle size.

Samples AO1-4 have been prepared by dropwise addition of an alcoholic solution of appropriate concentration of $AlCl_3$ to a glass vessel containing suitable amount of ethanol and ammonia. The average particle size of the powder obtained by this way was in nano scale (from 7-60 nm). However, the sample labeled $AO_{rev}$ has been prepared by altering the way of the reactants addition so that the ammonia has been added gradually to an alcoholic solution of $AlCl_3$. In this way, micro-sized particles of $Al_2O_3$ were obtained compared to sample AO1 which was prepared from the same reactans' concentration, see Tbable 1. The reason behind this is as follow: when adding an amount of ammonia to an alcoholic solution of $AlCl_3$, significant number of tiny particles are first nucleated in the initial high concentration of $AlCl_3$ at the induction period, then these sub-particles are accumulated to form very large and dense particles. Any other addition of ammonia to the reaction vessel after this period results in formation of other tiny particles that are consumed in the growth of the older particles [33]. In addition to that, in preparation of sample $AO_{rev}$, the concentration of $AlCl_3$ seems to be higher than that of sample AO1 because all $AlCl_3$ molecules are in the reaction vessel. Thus, at high concentration of $AlCl_3$ the rate of hydrolysis increases and then the consumption rate of sub-particles through condensation reaction will also increase [34]. All of these would consequently reduce the nucleation period, and hence decrease the total number of the formed nuclei which in turn produce $Al_2O_3$ particles with particle size in micro scale.

## 6. CONCLUSION

In this study, aluminum oxide nanoparticles have been synthesized via sol-gel process and from the hydrolysis of the alcoholic solution of $AlCl_3$ in presence of ammonia as a base catalyst. The effects of initial concentrations of catalyst ($NH_3$) and water on the particle size of synthetic nano alumina have been studied. The nanoparticles produced are spherical in shape, and their particle sizes are influenced by the concentration of both ammonia and water, and it was found that the particle size

increases as the concentration of these reactants increases. The obtained data are interpreted in terms of the dependence of the rate of hydrolysis and condensation processes on the initial concentration of the reactants which in turns affects the relative contribution of nucleation and growth steps. Increasing the hydrolysis rate tends to produce less number of nuclei during the nucleation process which ends with particles with larger size. Furthermore, altering the sequence of reactants addition has been also affected the particle size of the obtained alumina.

## ACKNOWLEDGMENT

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## العوامل المؤثرة فى حجم جزيئات اكسيد الالمونيوم النانوية المحضرة بطريقة السول – جل

تم تحضير دقائق أكسيد الألومنيوم النانوية عبر طريقة سول/جيل و ذلك من خلال تفاعل محلول كحولى لكلوريد الألومنيوم مع الماء فى وجود محلول الأمونيا كعامل حافز، وقد تم تغيير تكيزات الامونيا والماء وذلك للتحكم فى حجم الجسيمات النانوية الناتجة وقد تم التحقق من الجسيمات النانوية لاكسيد الالمنيوم التى حصلنا عليها من خلال التحليل الطيفى للأشعة تحت الحمراء وحيود الأشعة السينية وكذلك تم تحديد حجم الدقائق بواسطة المجهر الإلكترونى النافذ عالى الدقة، وقد ظهر أن الجسيمات النانوية لاكسيد الالمنيوم ذات شكل كروى و تتراوح احجامها من ٧ نانومتر إلى ٥٠ نانومتر و ذلك تبعا لتركيزات المواد المتفاعلة، كما اوضحت نتائج البحث أن زيادة تركيز الأمونيا والماء أدى إلى زيادة حجم الجسيمات كما انه بتغير إضافة المواد المتفاعلة بحيث يضاف الامونيا الي محلول كلوريد الالمنيوم ينتج جسيمات لها حجم كروى فى حدود الميكوميتر، كما عرض ميكانيكية التفاعل والتى من خلالها يمكن تفسير تأثير العوامل المؤثرة على حجم الجسيمات المنتجة.