

Ministry of Higher Education and Scientific Research

University of Baghdad

Institute of Laser for Postgraduate Studies



**Study optical limiting behavior and nonlinear optical properties for Silver nanoparticles in colloid and suspension using self-defocused technique**

A Thesis

Submitted to the Institute of Laser for Postgraduate Studies,

University of Baghdad

in Partial Fulfillment of the Requirements for

the Degree of Master of Science in Laser/ Physics.

By

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بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ

وَعَلَّمَ آدَمَ الْأَسْمَاءَ كُلَّهَا ثُمَّ عَرَضَهُمْ عَلَى الْمَلَائِكَةِ فَقَالَ

أَنْبِئُونِي بِأَسْمَاءِ هَؤُلَاءِ إِنْ كُنْتُمْ صَادِقِينَ {31} قَالُوا

سُبْحَانَكَ لَا عِلْمَ لَنَا إِلَّا مَا عَلَّمْتَنَا إِنَّكَ أَنْتَ الْعَلِيمُ

الْحَكِيمُ {32}

صدق الله العظيم

سورة البقرة ..... الآية {31,32}

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## **Acknowledgments**

At first, I thank **ALLAH** for everything because it pleased me things and make it easy. I would like to give special thanks to my supervisor **Asst. Prof. Dr. Mohamed K. Dhahir** for suggesting this work, his guidance, encouragement and support have definitely had a substantial impact on this work. I have learned experimental techniques. Special thanks to **Dr. Hussein A. Jawad** Dean of the Institute of Laser for Postgraduate Studies, for his continuous support and crateful care during the period of search. I would like to express my gratitude and thanks for **Dr. Zainab F. Mahdi** for allowing me to use the chemical Lab for my chemical experiment. I would like to present my appreciation **MS.Sally Abd Alrazzaq** and **Mr.Munaf Kassim** and for their continuous support and their endless help through my research work. Finally, I would like to thank my dearest people (**My family**), whom they had the greatest role to accomplish this work by their continuous encouragement and prayers. Hence I wish to dedicate this work to **my parents** for their love, encouragement, concern and moral support they have shown on me throughout my life, and do not forget my brothers (**Ali and Yasser**) for their continuous support and encourage throughout this work.

**Shahad Al.ani**

## Abstract

In this thesis the phenomenon of optical limiting effect of nanomaterials was studied by used defocused technique. Two types of lasers were used (blue laser 473 nm) and (green laser 532nm). In addition to the use of two types of nanomaterial's (AgNP<sub>s</sub> colloid) that have particle size (17\_27)nm and (AgNP<sub>s</sub> suspension) that have particle size ( 27\_ 44)nm to study the effect of optical limiting for them, deionized water was used for both as a solution. Two concentrations of diluted silver collide were made (31 and 11) ppm and diluted concentrations of silver suspension (29and 19) ppm. Where the behavior of the optical limit of silver colloid was first studied and shows the possession of the mentioned material threshold optical limit at power density (633\_638)W/cm<sup>2</sup> at concentration( 31 ppm 11 ppm)receptively when using blue laser ,and also at green laser at threshold power density(238\_240) W/cm<sup>2</sup>at concentration (31 ppm and 11ppm) receptively .Silver suspension exhibited optical limiting behavior when used blue laser at threshold power density (300 -308) W/cm<sup>2</sup>at concentration (29ppm and19 ppm)receptively , While the blurring of non-linear diffraction rings at green laser , Several tests were used such Tem , SEM , part per million (ppm), FTIR and (uv\_vis) measurement Furthermore, the non-linear refractive index and maximum change in nonlinear of the diluted concentrations of silver collide and silver suspension was calculated and graphed the relationship between the laser intensity that used and the number of diffraction rings.

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## List of Symbols and Abbreviations

Symbol	Meaning	Unit
$\Delta n_{nl,max}$	Maximum change of nonlinear refractive index	-
$L_{material}$	Thickness of sample	<i>cm</i>
$N_{rings}$	Number of rings	-
$\lambda_{beam}$	Wavelength of laser beam	<i>nm</i>
$Ag Np_s$	Silver nanoparticles	-
<b>SSPM</b>	Spatial Self- Phase Modulation	–
<i>cw</i>	Continuous Wave	-
<b>DPSSL</b>	Diode Pumped Solid State Laser	-
<i>E</i>	Electric field	<i>V/m</i>
$\epsilon_0$	Vacuum permittivity	<i>F/ M</i>
<i>I</i>	Intensity of laser beam	<i>W/cm<sup>2</sup></i>
$w_m$	The material weight	<i>g</i>
$w_s$	The weight of the solution	<i>g</i>
<i>N</i>	Number of microscopic dipoles pre unit volume	-
$n_2$	Nonlinear refractive index	<i>Cm<sup>2</sup>/W</i>
<b>NLO</b>	Nonlinear optics	-
<b>nm</b>	nanometer	-
$n_o$	Linear refractive index	-
<i>P</i>	Electric polarization	-
<b>P</b>	Dipole moment	
$P_L$	Linear polarization	<i>C/m<sup>2</sup></i>
$P_{NL}$	Nonlinear polarization	-
<b>DLVO</b>	Derjaguin, , Landau, Verway and Overbeek theory	-
<b>TEM</b>	Transverse Electromagnetic Modes	-

<b>Symbol</b>	<b>Meaning</b>	<b>Unit</b>
$AgNO_3$	Silver nitrate	-
$NaBH_4$	Sodium borohydrate	-
$pvp$	Polyvinyl pyrrolidone	-
$\theta$	Divergence angle degree	<i>degree</i>
$\alpha_o$	Linear absorption coefficient	$m^{-1}$
$\beta$	Nonlinear absorption coefficient	<i>Cm/Gw</i>
$\chi^{(1)}$	Linear susceptibility	-
$\chi^{(2)}$	Second nonlinear optical susceptibility	-
$\chi^{(3)}$	Third nonlinear optical susceptibility	<i>e.s.u</i>
<b>Wt. %</b>	Weight fraction	-
$V$	The volume of solution	<i>ml</i>
$M.wt$	The molecular weight of material	<i>g/mol</i>
$M$	The molarities.	<i>mol/L</i>
$wt$	The weight of the material	<i>g</i>
$TEM_{00}$	Gaussian beam	-
$UV-Vis$	Ultraviolet- Visible	-

# **Chapter One**

*Introduction and Basic Concept*

## 1.1 Introduction:

The nonlinear optics has opened the horizon in many scientific applications and technology [1], when Franken discovered in 1961 the second harmonic generation [2]. So this field has been used in many nonlinear applications such as Kerr effect, four wave mixing, self-defocused technique and other various applications [3]. Materials that have a nonlinear refractive index are usually used in nonlinear optical devices for example optical limiting and so on [4] [5]. The Nonlinear optics of the third degree occupies a large space in nonlinear optics [6]. Self-diffraction occurs in nonlinear optical materials when exposed to high power of light [7]. This process can be used to study the refractive index of nonlinear materials and understand the optical properties of these materials [8]. Much attention in the phenomenon of self - diffraction technique has become an effective tool for calculation of optical limits for the protection of optical sensor [9]. The non-linear refractive index can be calculated using several techniques such third harmonic generation, degenerate four waves mixing, z-scan technique and defocused technique [10]. The latter is one of the most widely used methods because of its advantages such as high sensitivity, simplicity, quick advantages and others [11]. To clarify more about the concept of non-linear optics it can be defined as the behavior of light when passing in non-linear materials [12]. Where the polarization depends on the non-linear intensity of the beam of light falling and this is what happens in high intensities [13].

## 1.2 Nonlinear optical effects

To describe how a non-linear phenomenon occurs one need to represent the passage of light through the nonlinear material. The nuclei and electrons attached to it create electric dipoles. The electric field of light will react with electric dipoles causing its oscillation. Therefore, these electric dipoles behave as sources of electromagnetic radiation [14]. Figure (1.1) shows how to separate the connected electrical charges by exposing them to a high light field to an electrically insulating material. This separation of charge results the formation of dipole moments  $P$ ; these vibrates incrementally as the optical field changes. The electric polarization  $P$  can be defined as the rate of electric dipoles in per volume the equation is described below [15]:

$$P = N \mathbf{P} \quad (1.1)$$

Where  $N$  is the number of electric dipoles per unit volume, and  $P$  is the dipole moment [16, 17].

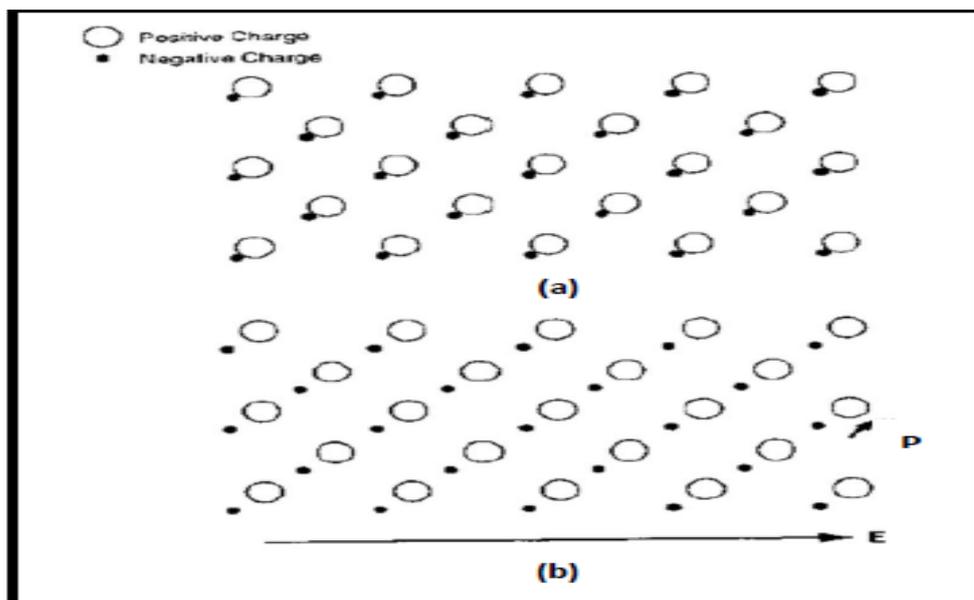


Figure (1.1): The response of a dielectric medium to an applied electric field. (a) Without field applied; (b) Field applied [18].

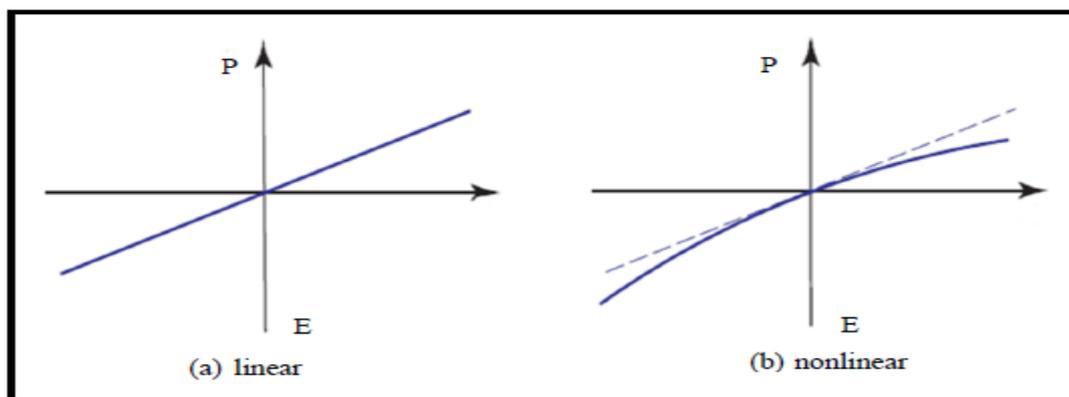
When exposed high power laser on the material this will lead to changes in the optical constants of the material (the absorption factor or the refractive index) of the material. The nonlinear term is derived from the polarization ( $P$ ) of matter that occurs by the optical field ( $E$ ) [19].

$$P = \epsilon_0 \chi(1) E + \epsilon_0 \chi(2) E^2 + \epsilon_0 \chi(3) E^3 + \dots ] \quad (1.2)$$

$$P = \epsilon_0 [ \chi(1) E + \chi(2) EE + \chi(3) EEE + \dots ]$$

$$P = PL + PNL$$

Where  $\epsilon_0$  is the vacuum permittivity and  $\chi_i$  is the linear susceptibility. The first term always represents linear polarization, while other terms represent nonlinear polarization. The mounts  $\chi(2)$  and  $\chi(3)$  represent the second and third order nonlinear optical susceptibilities, respective [ 20]. The relationship between  $P$  and  $E$  is a linear relationship as long as the values of  $E$  are approximated to values to inter-atomic electric field, which almost about  $10^5 - 10^8$  V/m [21]. Figure (1.2) represent the connection between  $P$  and  $E$  for linear and nonlinear insulator medium [22].



(a) linear

(b) non linear

**Figure (1.2): Shows ( $P-E$ ) relationship for (a) a linear dielectric medium, and (b) a nonlinear medium [22]**

In general, changes in the optical properties of the materials due to the high intensity of the falling light can be divided into changing the refractive index of the material or changing the absorption coefficient of the material. In case the absorption coefficient changes, the change can be described by the equation (1.3) [23]

$$\alpha = \alpha_0 + \beta I \quad (1.3).$$

Where  $\alpha_0$  is coefficient of linear absorption, while  $I$  is the intensity of optical field and  $\beta$  represent nonlinear absorption coefficient either the refractive index of the material can be described in the equation (1.4):

$$n = n_0 + n_2 I \quad (1.4).$$

Where  $n_0$  is the linear refractive index while  $I$  is the intensity of the light and  $n_2$  represent a nonlinear refractive index coefficient.

This coefficient has a clear effect on nonlinear optical phenomena such optical limiting, self-phase modulation, and self-diffraction [24, 25, and 26]. The nonlinear phenomenon can divide into many types as follow [27]:

### **Second-Order Effects**

1. Second harmonic generation (SHG).
2. Optical parametric amplification (OPA).
3. Pockle's effect.
4. Electro-optical beam deflection.
5. Optical rectification.

### **Third-Order Effects**

1. Third harmonic generation (THG).
2. Kerr effect.
3. Self-focusing.
4. Self-diffraction.
5. Self-phase modulation.
6. Solitons.
7. Four-Wave mixing (FWM).
8. Stimulated Brillion scattering (SBS).
9. Optical phase conjugation (PC).

### **And Higher-Order Effects**

#### **1.3 Third-Order Effects:**

Third order optical nonlinearities have the term it is called nonlinearity Susceptibility tensor and represent by ( $\chi(3)$ ). This term controlled optical Kerr effect, third harmonic generation and Brillion scattering .  $\chi(3)$  is generally linked to four frequency compounds, which are three fields interacting with one another to produce a fourth field. So the third order nonlinear based on the  $\epsilon_0 \chi(3) E^3$  in the polarization equation (1.2) [28,19]. In general, the nonlinear process of the third degree involves the interaction of waves containing four frequency compounds. These compounds, in general, may be similar to each other or some of them may be zero [29]. Third order nonlinearities have been studied in of manufactured materials such as polymer, nano-particles, homogeneous bulk glasses, metal and nano-crystals [30]. Nonlinear optical phenomena depend on the composition of new Frequency compounds and the

intensity of the light falling on the material that is working to change the coefficient of refraction [31].

### **1.3.1 Kerr effect:**

The optical Kerr effect was discovered in 1875 by Scottish physicist called John Kerr [32, 33]. It represents the change in the refractive index of the material with the change of the optical field [34]. Kerr effect, called also (ac Kerr effect), is a localized change of the refractive index of the material in a way that corresponds to the local beam of the falling light. This localized change of the refractive index of the material leads to several important phenomena like self-defocused, self-focusing and self-phase modulation.

### **1.3.2 Self-defocusing:**

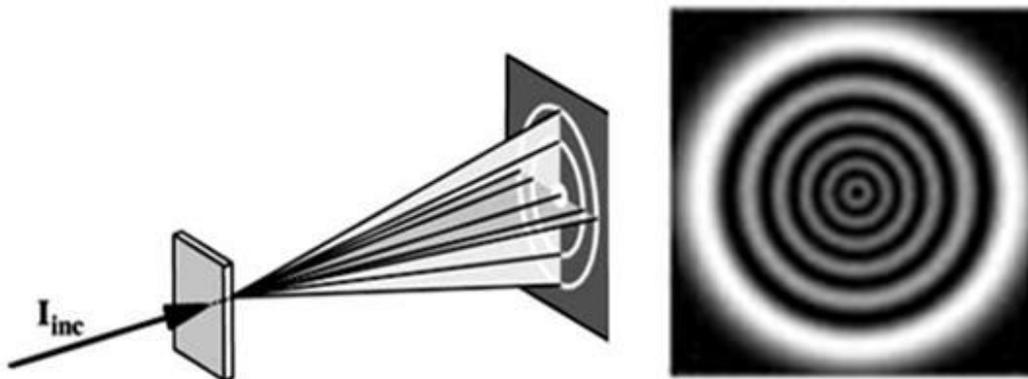
The interaction of the laser beam with the non-linear material results in a change in the refractive index of the material .This will produce spatial self-phase modulation for example spatial solitons. The most important of these processes is Self-diffraction (Self-defocusing)

This phenomenon is known in physics as an interaction between laser light and matter [35]. Diffraction rings (self-defocused) are formed as a result of the local change of the refractive index due to the local change of the optical intensity of the laser beam which will depart from its way as shown in the figure (1.3). The change in refractive index is proportional to the optical intensity falling on it. If the beam falling on the material is a Gaussian transverse, the change in the refractive index is also Gaussian. Through the calculation of diffraction rings of light so can know the greatest change in refractive index material [36]. As shown in equation (1.5). Diffraction pattern creation can be calculation by the Fruehauf integral. The phase of light emitted behind the material is

produced by the local delay in the sample, which is based on the refractive index of the material controlled by the intensity of the beam. As each phase shift ( $\pi$ ) causes a destructive interference, which in turn produces dark rings [37].

$$\Delta n_{nl,max} = \frac{\lambda_{beam}}{L_{material}} N_{rings} \quad (1.5)$$

Where  $\lambda_{beam}$  represent the wavelength of the light and  $L_{material}$  represent the thickness of the sample,  $N$  number of rings. In practice, the diffraction rings can be calculated in far field when a Gaussian beam is used to fall on nonlinear material [38, 39].



**Figure (1.3): the self-defocusing of a laser beam in a thin slice of matter results in a diffraction pattern behind the sample [39].**

Phase of beam delay after passage of the material and this represents the local delay of the path of light during the passage of the material and this indication of the local change of the refractive index of the material depending on the form of distribution of the intensity of the light [40]. On

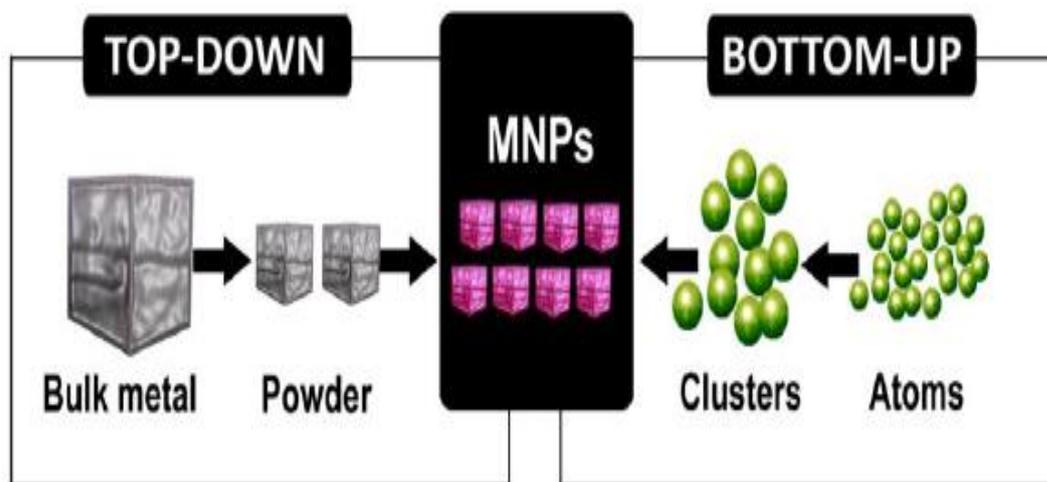
The other hand, in geometrical optics can be used self-focused to describe self-defocused because diffraction of light is also graded [41].

### 1.4 Optical limiting:

Nonlinear optical materials attract attention due to the large number of applications in several fields, including optical storage and optical limit. The optical limit is one of the most important applications as a result of its use in the protection of human eyes and laser sensors [42]. The optical limiting phenomenon can be obtained by several mechanisms based on nonlinear materials, like (induced scattering, self-focusing, induced aberration, two-photon absorption, and self-defocusing and photo refraction) [43]. The use of the optical limit in the laser, because of the ability of the optical limit to control the intensity of falling beam, when a laser beam falls on an object that has a particular optical limit, the intensity of the external beam of the material increases linearly with the increasing intensity of the falling beam, but at a certain intensity above (**threshold intensity**), the transmitted intensity of the laser becomes constant and the material become opaque [44]. The optical limit results from the fact that radiation depends non-linearly on the optical properties of the material [45]. In optical limiting often called material that possesses an optical limitation characteristic (**chromospheres**), bulk glasses are very favor when it used in optical limit devices while thin films are prone to damage, there are several substances possessing optical limit properties like organic and inorganic particles and metal nanoparticles [46]. The optical limit feature depends on several factors including the particle size, the cost and the concentration of non-linear material [47]. One of the benefits of optical limiting is the protection of non-linear materials from damage when subjected to high intensity of the laser [48].

## 1.5 Nano materials

Nano science is a science that is studied by particles that have dimensions within (1\_100) nanometers. This science has become one of the most important sciences at the present time because these material properties have unique properties and several applications [49]. Bulk materials have the characteristics of continuous physical as well as materials with small dimensions or known as micron sized such as sand grains. But in the case of Nano scale materials, the laws of classical physics do not apply to them such as energy, motion and others. But the laws of quantum physics is apply. For example, the silver material on the nano scale has properties (the optical properties and mechanical properties) that differ from those on the micro scale (bulk) [50]. When named it nanoparticles this means that the three dimensions of the material are on the Nano scale [51]. Nanomaterial's can also be formed by two methods, as shown in Fig (1.4)



**Fig (1.4):** Schematic shows the (top-down) and (bottom-up) approaches for the making metal nanoparticles [52].

### **1.5.1 Top-down approach**

In this approach, large objects are converted to sizes with Nano scale dimensions by traditional methods such as milling, shearing or others [53]. This process reduces the volume of materials (bulk) and is not suitable in the formation of regular forms, where the problem of irregular surfaces is one of the disadvantages of this approach and this has a significant impact on the physical properties and chemical properties of nanoparticles causing significant damage to the crystalline patterns of the resulting forms [54]

### **1.5.2 Bottom-up approach:**

The approach from bottom to top indicates the accumulation of materials from the bottom like molecule-by-molecule, atom by atom, cluster by cluster. This approach is the basic approach used for the formation of nanotubes of regular sizes, surfaces and distribution [55]. Colloid dispersion is one method used to form nanomaterials, an example of an approach from bottom to top. One of the advantages of this method is the composition of Nano materials less imperfect and more homogeneous chemical [56]. It is effective in the chemical composition of nanoparticles specifically which can control the growing of nanomaterials. It is also used effectively in processing and manufacturing nanostructures [57].

## **1.6 Silver Nanoparticles (AgNps):**

The (AgNPs) have become an integral part of human life. They are found in clothing and care products for humans because of their resistance to bacteria [58, 59]. Because of its unique physical and electronic properties, it has been used in many applications, medical applications such as skin care creams, feet, dressings for wounds, cosmetics and many examples.

To understand the shape and size of these nanoparticles of silver are important to improve their performance [60].

### 1.6.1 Synthesis of silver nanoparticles (AgNPs):

In general, (AgNPs) can be prepared using three main methods: physical, chemical and biological [61]. As shown in Figure (1.5). Each one of these methods has different efficiency than the other. Parameters such as shape, diameter, and stability are closely related to the method chosen for making nanomaterial's [62].

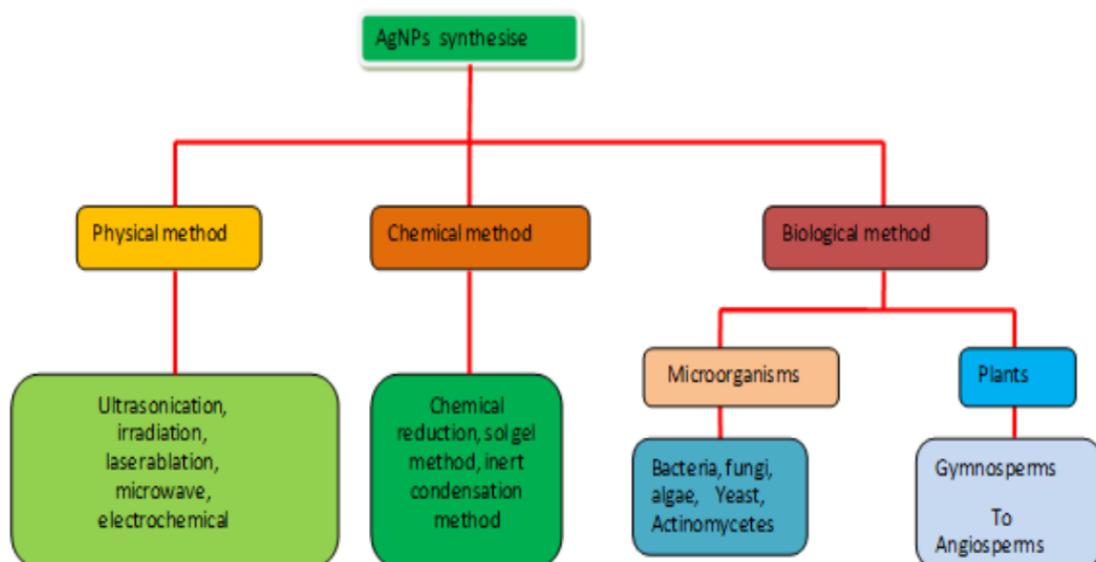
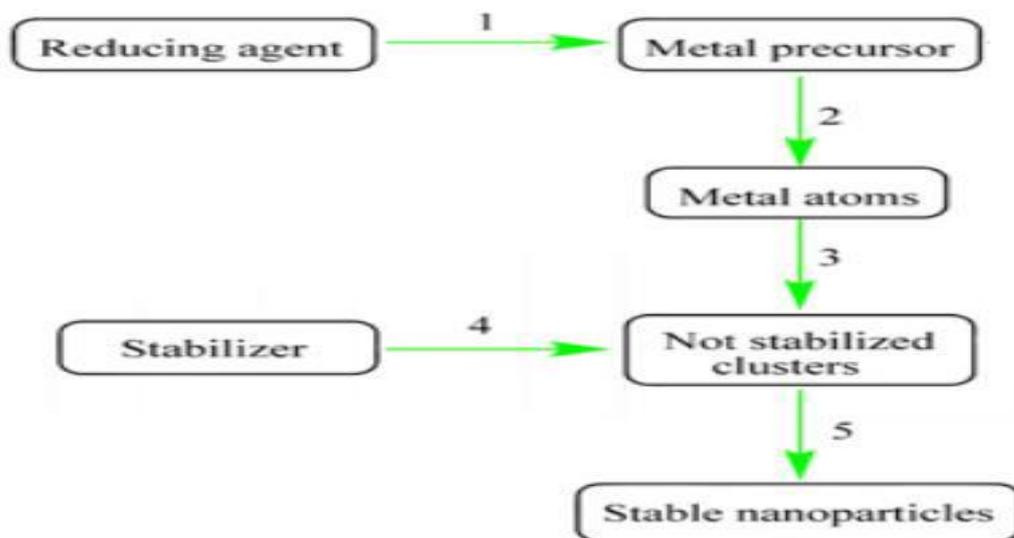


Fig (1.5): illustrates the methods of synthesise AgNPs[62]

### 1.6.2 The chemical method :

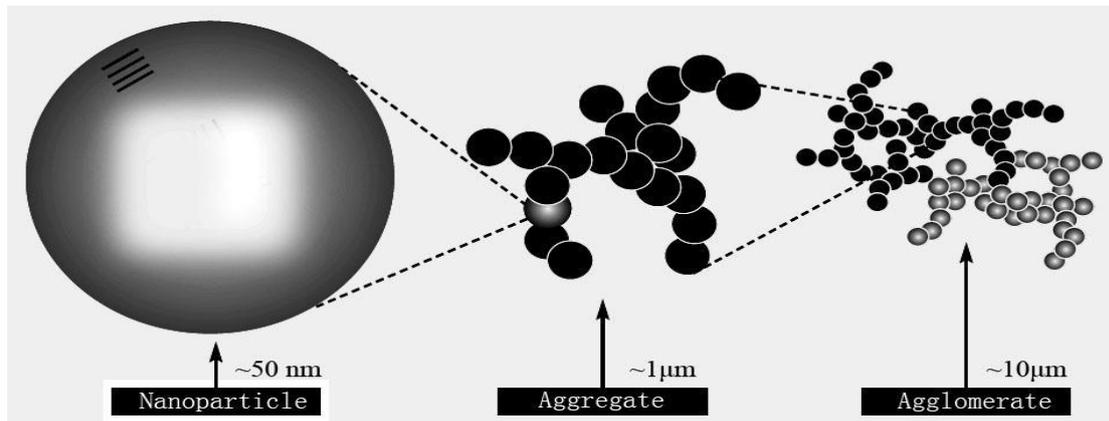
The method of chemical reduction is one of the ways to obtain (AgNPs) in its fixed form or on its dispersion fluid in water or in organic solvents. The reduction of silver ions in the solutions leads to the creation of silver colloidal which possesses the sizes of Nano. The reduction of different compounds with silver ions leads to the creation of silver atoms. This will be followed by aggregation into oligomer clusters. These clusters will

lead to the formation of silver colloidal particles [63, 64]. Figure (1.6) illustrates this method and is based on a bottom-up approach. It is based on the reduction of silver salt, (silver nitrate), which is widely used as a result of cheap price and has a chemical stability [65]. With strong or weaker reduction factors (borohydride[66]. Citrate [67]. ascorbate [68] . Hydrogen gas [69] .With an adequate amount of stability (sodium dodecyl sulfate [70].(polyvinyl pyrrolidone) [71,72]. polyvinyl alcohol [73]. Polymethylvinylether [74].



**Fig (1.6): Schematic shows suggest chemical reduction process; the numbers on the arrows shows the chronological arrangement of the operation steps [75].**

These particles can also stick together to form aggregates of increasing size due to attractiveness as shown in figure (1.7).

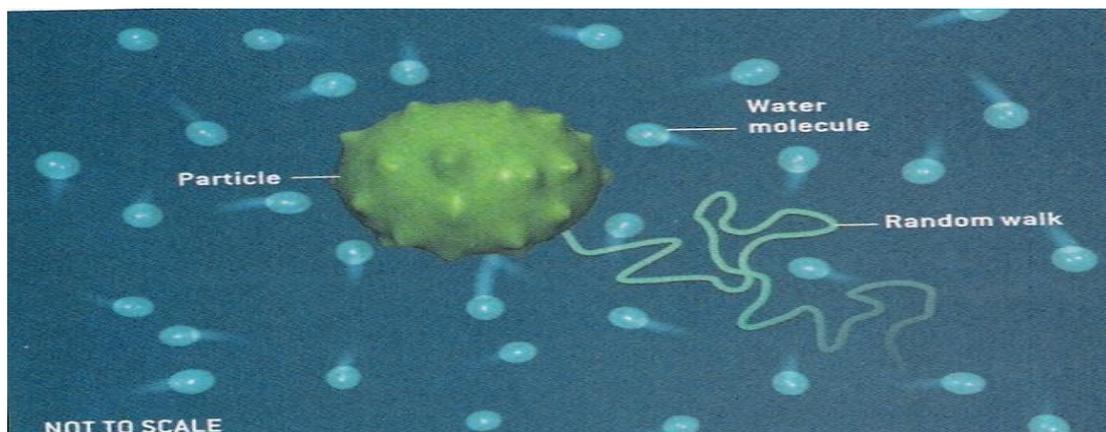


**Fig (1.7): shows the schematic diagram of the Nano-particles aggregation and agglomeration [76].**

The term stability means that the particles do not aggregate at a high rate. The agglomeration rate can be calculated by the number of collisions and the probability of adhesion during the collision. The theory (DLVO theory) was also developed for the calculation of colloidal stability by Derjaguin, Landau, Verway and Overbeek [77, 78]. This theory suggests that the stability of particles in a solution can be calculated through the sum of the attractive vander waals bonds and the electrical repellency forces present between the particles. If the force of attractive is greater than the repulsive force, these particles will become collide and then the suspension will become unstable. In the case where the high repulsion is dominant, the suspensions will be stable. In the case of steady of stable colloidal fluid the force of expulsion must be predominant. Mechanisms that influence the stability of colloidal material can be divided into two types, namely, steric repulsion and electrostatic repulsion. Electrostatic stability produces an electric double layer surrounded the nanoparticle. This layer causes the columbic repulsion between nanoparticles. In the case of steric stabilization, an example of that polymer Polyvinylpyrrolidone (pvp) produced by the adhesion of molecules of polymer along the nanoparticles [79, 80].

## 1.7 Nano-fluids and Brownian motion:

Nano fluid is the distribution of Nano scale materials (nanotubes, nanoparticles, or Nano fiber) in basal fluids. And can define Nano fluid as Colloidal material or suspensions containing nanoparticles. Nano fluids have been used because they possess unique physiological properties such as thermal conductivity, viscosity, thermal diffusion and other properties compared with the primary fluids [81]. Nano fluids also show more complex heat connections compared to solvents [82, 83]. Brownian motion is the random movement of particles in a Nano \_fluid [84].as show in figure (1.10).



**Fig (1.8): Brownian motion [85].**

It is called the Brownian motion back to its discoverer, the scientist Brownian, who used a microscope to see the pollen in the water, where this phenomenon was explained by(Einstein in 1905), where he explained this movement of pollen grains by moving random water molecules, which is responsible for pollen movement. This indicates the presence of molecules and atoms and therefore enhanced particle theory [86]. The energy in the liquid is transferred in four ways: thermal conductivity, conductivity in the base fluid, micro-convection caused by random movement of nanoparticles and collisions between nanoparticles [87]. Increasing the thermal conductivity of the Nano \_fluid is associated with

the small size of the particle inside it because the particles of smaller size have high a random movement (Brownian motion) [88].

### **1.7.2 The properties of Nano fluids:**

Nanomaterials are distinguished by their characteristic thermal properties such as thermal diffusion and thermal conductivity [89]. The nanoparticles have these distinguishing features compared to other materials because of it have a surface area to volume ratio due to the presence of several atoms on the border and this makes them more active in suspension [90]. The nano suspension is highly conductive because of the convection motion between the liquid surfaces and the solid particles. The nano suspension has a high thermal conductivity compared to the base fluid. Improve the thermal properties of nanoparticles due to the small particle dimensions. Nanomaterials have improved properties than other materials of micro meter size or millimeter such processes related to erosion, sedimentation, blockage [91]. The viscosity is characteristic in the liquids of the nanoparticles because of their effect on the transfer of heat between the components of the liquid, which in turn depends on the basic viscosity of the fluid added to the concentration of nanoparticles. Scientific studies have shown that there are several factors affecting the viscosity of nanoparticles such as particle diameter and temperature. In addition to several factors such as volumetric concentration of nanoparticles, pH and particle form [92].

## **1.8 Laser source:**

Laser is a powerful source of light and has several qualities and features not found in ordinary light sources for example mercury lamps, laser light has the ability to light travel over long distances without the occurrence of diffraction or dispersion of light [93]. There are several types of laser, including liquid laser, solid laser (semiconductor lasers), gas lasers and other types, but all of them share a distinctive element is the presence of material to amplify the radiation [94]. Therefore, the laser is used in several different applications; the one feature of the laser is its possession monochrome and excellent directional [95]. The laser beam can be described as electromagnetic waves where the magnetic and electric field is vertical on the direction of propagation of the wave [96].

### **1.8.1 Diode Pumped Solid State Laser (DPSS):**

Diode pumping is the simplest way to pump the active medium. In light of the evolution of science and technology, diode pumping is now dominant despite the high cost of the diode pumping but it produces less heat and the high efficiency than others laser[97] . Figure (1.12) shows laser system (*DPSS*).

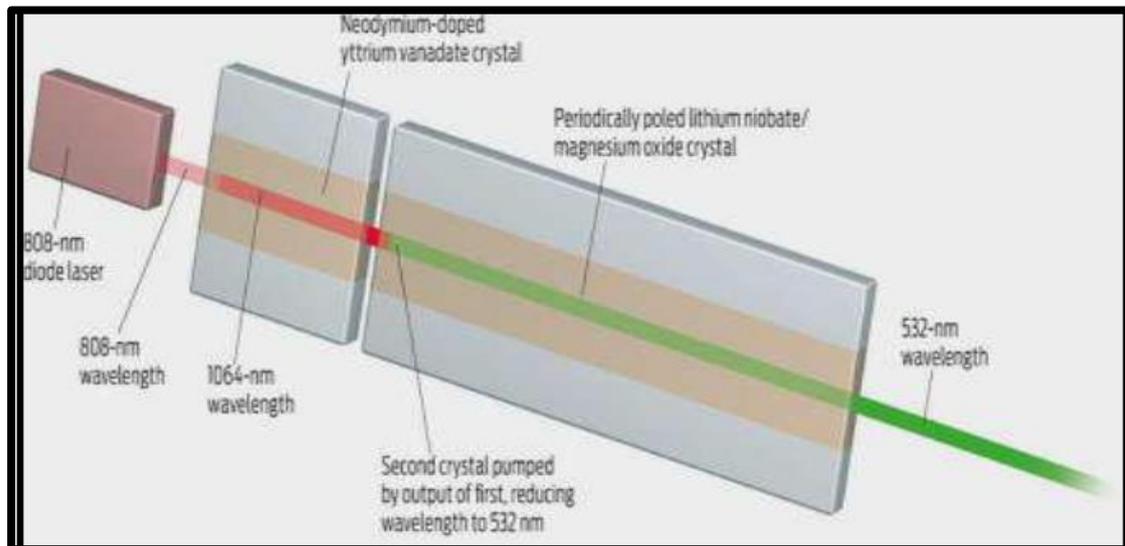


Figure (1.9): laser system (DPSS) [98].

### 1.8.2 Advantages of Diode Pumping:

1. Reducing (noise) when using diode pumping in diode lasers
2. The operating lifetime of these brands of laser is much greater than that of traditional lamps, often above (10000) hours
3. Reduce the thermal load of the crystal because these laser diodes pump a very narrow optical beam proportional to the absorption package suitable for laser action.
4. Also, diode pumping can be used in several solid state lasers

### 1.8.3 Features of diode laser beam:

The laser is a light source that exhibits unique properties like monochromatic, coherence and directionality [100]. It is used in many fields like medical, engineering and industrial applications in addition to scientific research fields

One of the most important features is: [101].

1. Riley length
2. Transverse electromagnetic mode
3. Beam diverge

#### 1.8.4 Gaussian beam, beam diameter and spot size:

The Gaussian beam possesses the highest concentration of the falling beam. It is the ideal beam used [102]. As shown in fig (1.14) [103]. The diameter of the beam can also be defined as the distance by which the amount of energy is reduced by  $(1/e^2)$  of its maximum energy value ( $1/e^2 = 0.135$ ). The region within this circle contains a total of ( 86.5% ) of the total energy value of Gaussian beam [104] .The spot size of the Gaussian beam (one half of beam diameter) can also be defined as the distance from the center point of maximum power to the point where the energy is reduced to  $1/e^2$  [ 105].

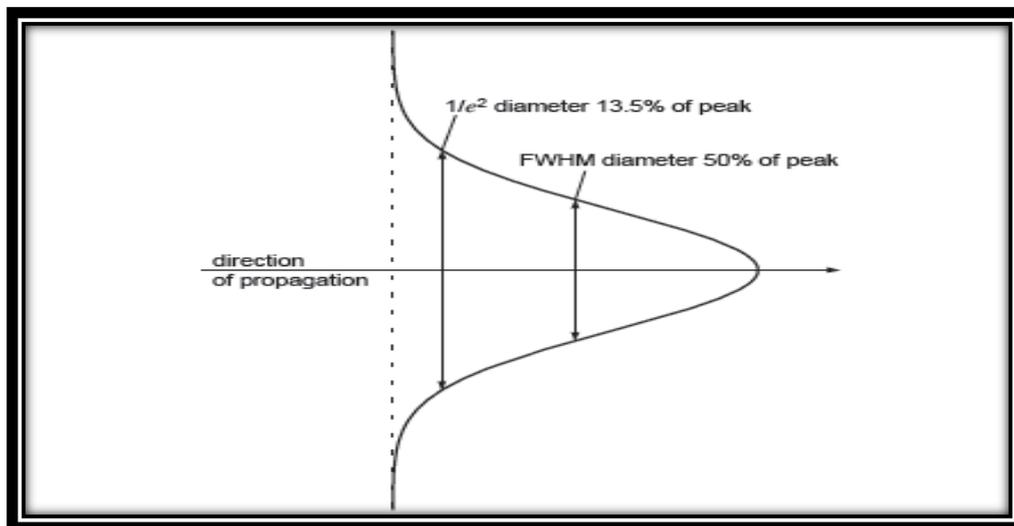


Fig (1.10): profile of Gaussian beam [103].

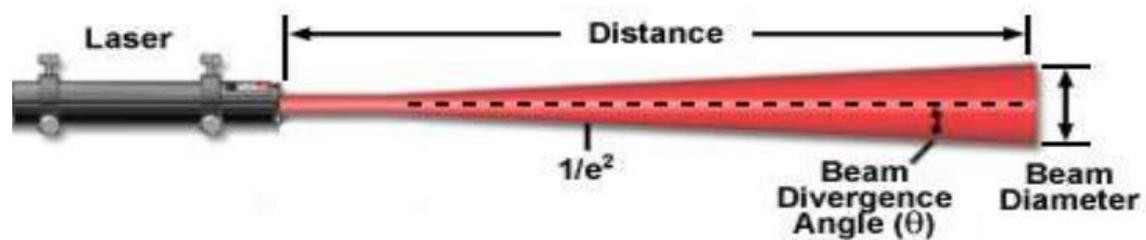
#### 1.8.5 Beam divergence:

Light diffuses or disperses when traveling long distances Laser is one of the sources of light which is characterized by a high direction, but may be scattered by a small proportion of light during the transition to long

distances as shown in fig (1.15) .The angle of dispersion can also be described by the symbol ( $\theta$ ) as in the equation (1.6)[ 106].

$$r_s = f \theta \quad (1.6)$$

f: refer the focal length of lens  $r_s$ : refer the spot diameter at focal point



**Fig (1.11): laser beam divergence (106)**

## 1.9 Literature survey:

In 2006, Chávezarda and et al computed far field patterns from the interaction of the laser beam with defocusing material, where a continuous laser beam was used and dropped to a colloidal material have a large nonlinear refractive index; their results were consistent with the self-defused medium theory based on the Kernshov diffraction [107].

In 2008, Guzman and et al prepared nanoparticles by means of chemical reduction. Silver nitrates were used as metal precursors and hydrazine hydrate as a reducing agent. The nanoparticle dimensions were measured about 60 nm , these particles were highly affected against macrophages and gram positive bacteria called E. coli and Staphylococcus aurous against methicillin [108].

In 2010, Ara and et al observed non-linear optical parameters of –silver nano particles using a scanning technique. Helium Neon laser with a

wavelength of 632 nm was used at different intensities. These particles were prepared by reducing silver nitrate by hydrogen gas. The process was performed at 70 ° C, the preparation was for 3 hours and the sizes of these particles up to 20 nm and measured non-linear refractive index it limits of (7-10) ( $\text{cm}^2/\text{W}$ ) with a negative sign and this refers to the defocused technique in silver nano particle[ 109].

In 2012, R. Zamiri and et al managed to achieve the theory of spatial phase modification (SSPM) for palm oil containing silver nano particles. The solid state laser was used for diode laser at a continuous wavelength of 405 nm at 50 mW. Note that self-diffraction patterns indicate a non-linear refractive index. These patterns are increased by increasing the non-linear refractive index of the material and the patterns have been significantly correlated with previously explored patterns [110].

In 2013, Ammar Mohamed Taemeh studied the non-linear refractive index of a liquid dye by self-defocused technique using a 532 nm diode laser and studying the effect of temperature on the different concentrations of dye material at different laser intensity and calculation of refractive index change Non-linear due to the effect of heat on liquid dye concentration [111].

In 2015, Y. Zhang and et al calculated the far – field patterns of the atomic complex Rb, and found that these patterns are strongly correlated with the intensity, frequency, atomic number and sample position. In addition, these patterns reflect the sensitivity of nonlinear optical properties. The results obtained are of significance in many phenomena. Such optical limiting and nonlinear physic [112].

In 2015, S. Xiao and et al were able to study the self-diffraction pattern of the  $\text{MoS}_2$  supernatant solutions and conduct the dynamic simulation of data and analysis. Results showed that diffraction patterns can be

divided into three stages. In the first stage, the Gaussian laser beam changes to similar diffraction patterns in the second stage obtain similar symmetric rings. In the third stage, ring become asymmetric horizontally as a result of the laser structure [113].

In 2016, each of the Krishnakumar and et al created silver nano particles by two methods using tri-sodium citrate as a reducing agent for silver nitrates. Different sizes of nanoparticles were obtained by continuous heating and periodic heating. The (uv-vis) technique revealed that the greatest absorption of particles was at 450-460 nm wavelengths, the non-bound form of these particles has a size of 60 - 80 nm [114].

In 2016, Manaf Kassem Khalaf Institute of Laser University of Baghdad studied the non-linear behavior of the –silver nano partial and multi-walled carbon in acetone solution and in deionized water using a different intensity of laser beam with a wavelength of 532 nm [115].

In 2017, Sally Abdul Razzaq Institute of Laser University of Baghdad to prepare particles of silver nano in multiple sizes in a short time and cost less and explain the properties of physical and quantitative addition to study the behavior of non-linear of those particles by the technique of self de focused [116].

In 2018 Sultan.H A and e t al From the University of Basra, College of Science, study non-linear optical properties of the solution of dimethoxine Kurcumin in a multi-diffraction rings, a Continuous laser was used during the solution and the non-linear refractive index was calculated in addition to the study of the optical limit of this solution at a wavelength of 437 nm and was an experiment with results matching the previous research [117].

In 2019 Akram Shaker and et al Institute of Laser University of Baghdad In this work, a nonlinear characteristic of Nano fluid consisting of single-

walled tubular carbon and multi-walled tubular carbon is suspended in fluid bases in two fraction sizes. The results showed that the number of diffraction rings observed indicates the maximum change in the nonlinear refractive index of plankton detected by CCD. The results of this work showed that the largest change in the nonlinear refractive index ( $\Delta n_{nl, \max}$ ) increased by either the intensity of the incident laser or the fractional size[118].

### **1.10 The aim of work:**

1-Study the optical limitation properties of colloid and suspension of silver Nano materials

2-make comparison study between them to reach the optimum condition of silver Nano particle condition of optical limiting Phenomenon

# Chapter Two

## *Experimental Details*

## **2.1 Introduction:**

This chapter contains a description of several topics, including the chemical part which contains the method and the chemicals used to form (AgNPs) colloid and suspension. Also Physical part that describe the system used to measure the optical properties and optical limiting of (AgNPs) in colloid and suspension by defocusing technique and also describe the physiochemical properties of silver nanoparticles(AgNPs)

## **2.2 The chemical part in (AgNPs) colloid:**

The chemical method one step method is used (bottom to up). Where the precursors were reduced by using reduction factor with addition of stabilized substance to form the nano colloid. The Precursors are the silver nitrate ( $\text{AgNO}_3$ ) with concentration (0.015)g ,the sodium Borohydride ( $\text{NaBH}_4$ ) with concentration (0.002)g , as reduction agent and the stable agent(prevent aggregation ) is Polyvinylpyrrolidone (pvp) with concentration (0.05) g . These substances have all been dissolved into deionized water (di) to create aqueous solution.

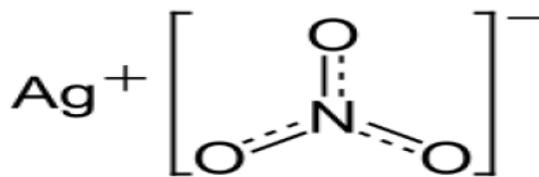
### **2.2.1 Materials used to prepare (AgNPs colloid):**

#### **2.2.1.1 Silver Nitrate:**

$\text{AgNO}_3$  (SIGMA CHEMICAL CO.). The molecular weight is (169.78)  $\text{g}\cdot\text{mol}^{-1}$ . Assay 99 %.As shown in the figure (2.1)



A



B

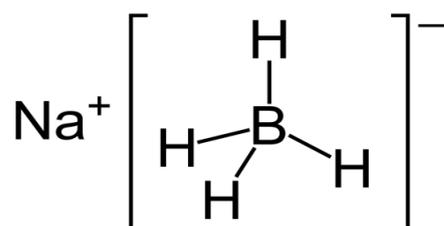
**Figure (2.1): A: Silver nitrate crystals. B: Chemical formula for silver nitrate**

### 2.2.1.2 Sodium Borohydride:

$\text{NaBH}_4$  (Chemical Point Germany). The molecular weight is (37.83)  $\text{g}\cdot\text{mol}^{-1}$ . Assay 99.9 %.As shown in the figure (2.2)



A



B

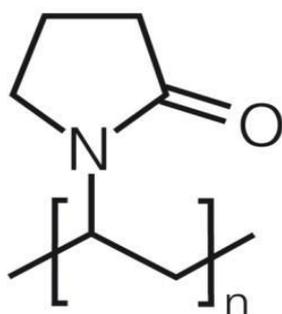
**Fig (2.2) A: Sodium borohydride lumps, 25 grams Wireframe model B: chemical formula of sodium borohydride**

### 2.2.1.3 Deionized water:

Deionized water is the purest water of distilled water, the water from which the ions have been removed

### 2.2.1.4 Polyvinylpyrrolidone :

(pvp) The molecular weight is  $(2.500) \text{ g}\cdot\text{mol}^{-1}$ . (Assay 99%) As showed in the figure (2.3)



A

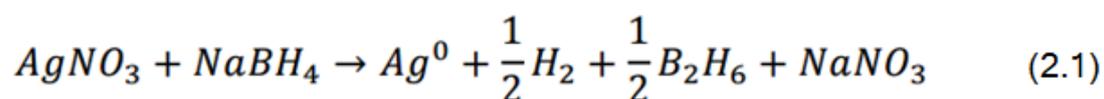


B

**Figure (2.3) A: Structure of Polyvinyl pyrrolidone  
B: Sample of polyvinyl pyrrolidone**

### 2.3 sample preparation:

The Silver nitrate ( $\text{AgNO}_3$ ) with concentration ( $0.002\text{g} / 20\text{ml}$  of Di water) was added with a fixed amount of Sodium Borohydride ( $\text{NaBH}_4$ ) with concentration ( $0.015\text{g}/15\text{ml}$  of Di water) and by using the pvp as anti-aggregation material with concentration ( $0.05\text{g}/5\text{ml}$  of Di water). A ( $\text{AgNPs}$ ) Colloid was obtained with certain properties using equation (2.1) [119]



The controlled condition such as maintaining the temperature of the solution at ( $30 \text{ C}^0$ ) used to prepare the  $\text{AgNP}_s$  Colloid

### 2.3.1 The concentration laws used to prepare AgNP<sub>s</sub> colloid:

1. Both the Weight of AgNO<sub>3</sub> and NaBH<sub>4</sub> were obtained by the following equation (2.2) [120].

$$M = \frac{wt}{m*wt} * \frac{1000}{V} \quad (2.2)$$

Where  $M$  refer to the molarities (mol/L), and ( $wt$ ) refer to the weight of material used in g, ( $V$ ) refer to the volume of solution (in ml). ( $m * wt$ ) Refer to the molecular weight of material (g/mol)

2. The weight of the PVP is calculated by the equation below (2.3) [121].

$$wt. \% = \frac{W_m}{W_m + W_s} * 100 \quad (2.3) \quad [121].$$

$W_m$  Refer to the material weight  $W_s$  while refer to the weight of the solution.

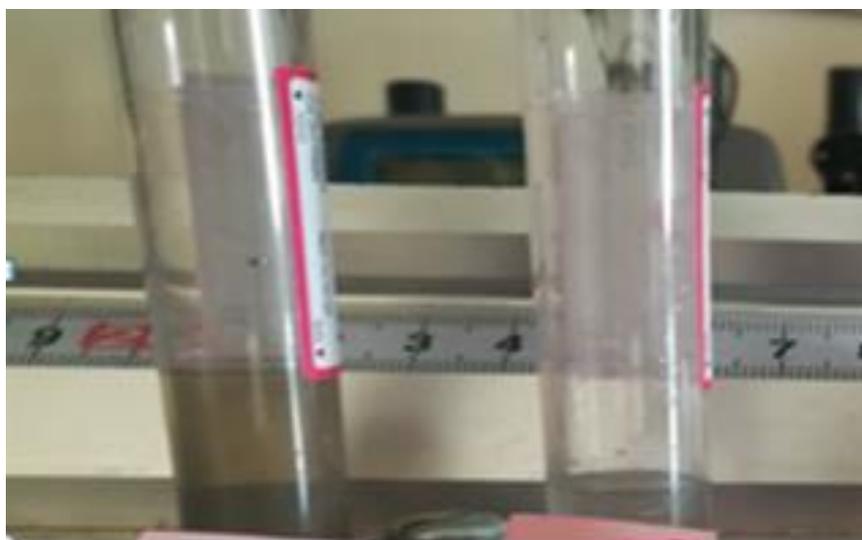
### 2.3.2 Steps of the experiment to form (AgNPs) Colloid:

1. To produce silver Nano particle a (0.002) g of NaBH<sub>4</sub> is placed in the beaker and (20) mL of deionized water is added over it
- 2- Place the mixture on a magnetic stirrer for 10 minutes with mix by using magnetic bar
- 3- Place 0.015 g of AgNO<sub>3</sub> with 15 ml of deionized water
- 4- Place the mixture on a magnetic stirrer for 10 minute
- 5- The NaBH<sub>4</sub> solution is cooled by ice cubes and the temperature is measured after 15 minutes by using a thermometer at (2C<sup>0</sup>). The purpose of cooling is to avoid high heat solution during reaction

- 6- The  $\text{NaBH}_4$  mixture is placed on a magnetic stirrer and an  $\text{AgNO}_3$  solution is added by borosilicate glass
- 7- Prepare a pvp mixture by adding 5 ml of deionized Water to 0.05 g of pvp and place the mixture on a magnetic stirrer
- 8 - A PVP solution is added to a mixture consisting of  $\text{NaBH}_4$  and  $\text{AgNO}_3$  gradually during the stirring process to prevent aggregation
- 9- The color of the solution gradually changes from yellow to black
- 10- Place the solution in several tubes and place it in a cool place

### 2.3.3 Preparation of diluted samples of AgNps colloid:

Two types of sample was prepared the first sample (type a) contain (4 ml of deionized water and 6 ml of  $\text{AgNP}_s$ ), the second sample (type b) contain (7 ml of deionized water and 3 ml of  $\text{AgNP}_s$ ) as shown in the figure (2.4) .



**Fig (2.4):** diluted samples of AgNps colloid

## 2.4 The chemical part in AgNPs suspension:

The chemical method for the preparation of a suspended silver material is through the use of silver nanoparticles (in powder form) with deionized water (as a solution) and the use of the magnetic stirrer to mix them.

### 2.4.1 Materials used to prepare (AgNPs suspension):

#### 2.4.1.1 The Silver nanoparticles (Ag NP<sub>s</sub>):

The use of silver nanomaterial's in the form of powder (Hongwu International Group Group LTd) with particle size (20nm) and the most important features of this powder are shown as in the table (2.1)

**Table (2.1): physical and chemical properties of silver nanoparticle [122].**

weight	10gm
Particle size	20 nm
Purity	99.9 %
Shape	spherical
Color	Gray black powder
Production	China

#### 2.4.1.2 Deionized water:

Deionized water is the purest water of distilled water, the water from which the ions have been removed

## 2.5 sample preparation:

Silver powder was weighted by a high precision electronic balance (type BL-210S, Germany). Where the equation was used to calculates the concentration of the solution by weight (*wt. %*)[121].

$$wt. \% = \frac{W_m}{W_m + W_s} * 100 \quad (2.3)$$

( $w_m$ ) Refer to the material weight, while ( $w_s$ ) refer to the weight of the solution .In addition, the magnetic stirrer device with magnetic bar was used to mix the solution well to obtain a homogeneous mixture (preferably mixing at least 2 hours)

### **2.5.1 Steps of the experiment to form (AgNPs) suspension:**

1. Take a certain amount of powder Nano silver
2. Put this amount in a certain volume of deionized water
3. The relative weight ratio of the suspension was calculated by the equation (2.3)
4. Using different weights of silver powder nanoparticles in a fixed volume amount of deionized water to obtain diluted samples of different concentrations

### **2.5.2 Preparation diluted samples of AgNps suspension:**

Two types of sample was prepared the first sample (type A) 0.02 Wt.% consist of ( 50 ml of Di water with 1.5 g of AgNP<sub>s</sub>), the second sample (type B) 0.009Wt.% consist of (50 ml of Di water with 0.5 g of AgNP<sub>s</sub> ), as shown in the figure (2.5)



A

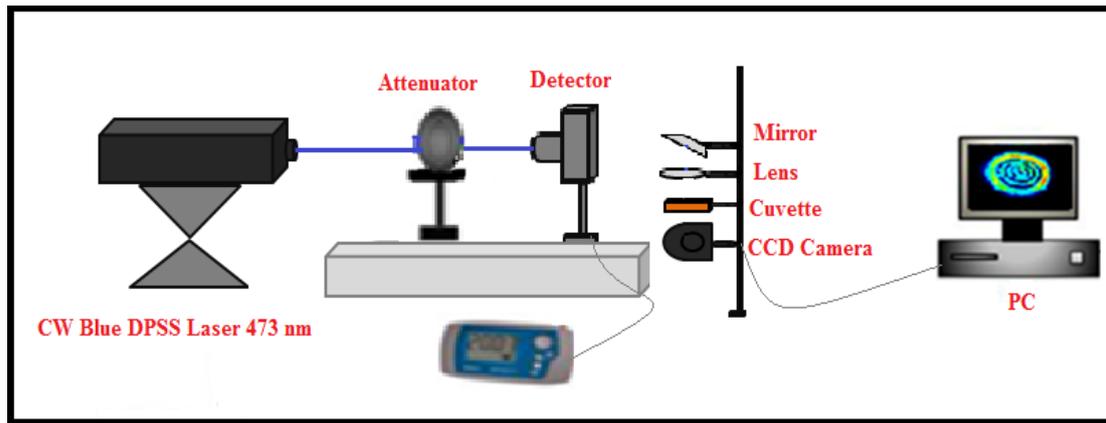
B

**Figure (2.5): The diluted samples of AgNPs suspension**

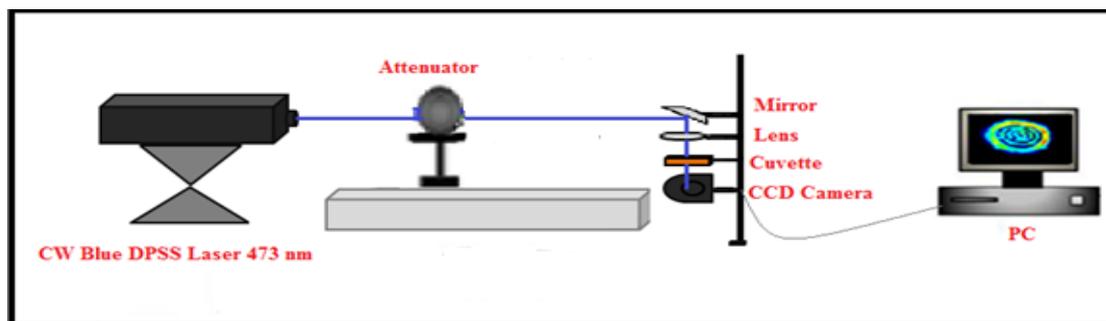
## **2.6 The Physical part:**

### **2.6.1 The experimental set-up:**

The two types of lasers were used; blue laser device with 473nm wavelength and the green laser device with 532 nm wavelength. By using the attenuation one can be control laser power that dropped on the cuvette (Thickens 5 mm) containing AgNPs colloidal or suspension. Multiple laser power measured by a laser power meter (UNO-200982, gentec-EO, Canada).By using the mirror the vertical alignment was obtained. The laser beam was focused by a lenses with a focal length of(7and5) cm in case AgNP<sub>s</sub> colloid and AgNP<sub>s</sub> suspension respectively .Through the ccd camera connected to the personal computer (pc) containing a program for the monitory of patterns of nonlinear diffraction. As shown in the figures (2.6, 2.7)



(A)



(B)

**Fig (2.6): Schematic diagram of the experimental set up (A) measuring the power. (B) The beam focusing on the sample**



**Fig (2.7): picture of the experimental set up**

## **2.7 Laser Source at 473 nm cw laser (blue laser):**

This laser is shown in the figure (2.8), (The model MBL-FN-473nm-

200mW), with wavelength at 473 nm (**cw Laser**). This laser is pumped by the diode laser



**Figure (2.8): (a) Laser source, (b) power supply**

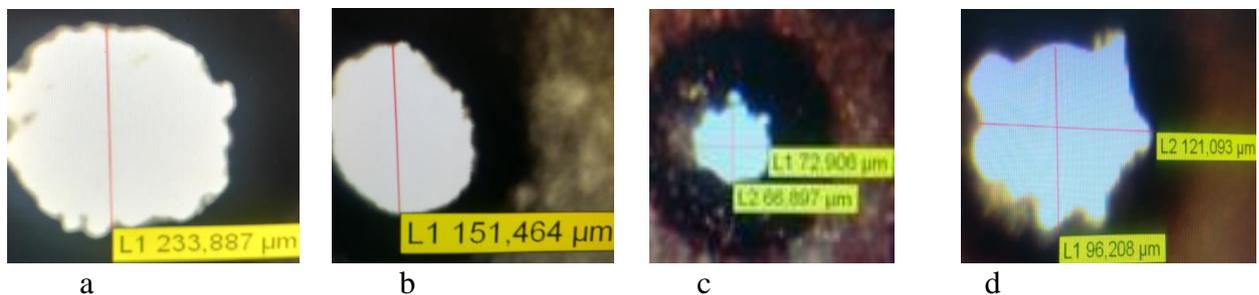
The following table (2. 2) shows the most important parameter of this laser (blue laser) [121].

**Table (2.2): Specifications of the blue laser [121].**

Output Power	299.1 mW
Power Stability over 4 hours	1.39%
Operating Mode	CW
Transverse Mode	TEM00
Beam Diameter ( $1/e^2$ )	1.751 mm 1.412mm
Beam divergence( full angle)	1 mrad

## 2.8 calculated laser beam diameter:

Diameter of laser beam was measured directed to the material by the microscope (model ME.2665, EUROMEX – HOLLAND), Where the thermal paper was placed instead of cuvette and measuring the diameter of this hole by the microscope was (233,887  $\mu\text{m}$ ) for green laser(532nm) and (151,464  $\mu\text{m}$ ) for blue laser(473nm) in case  $\text{AgNP}_s$  colloid, and diameter of hole was (105  $\mu\text{m}$ ) for blue laser and was (169,197  $\mu\text{m}$ ) for green laser in case  $\text{AgNP}_s$  suspension as shown in the figure (2.9)



**Fig (2.9):**the diameter of green laser in  $\text{AgNP}_s$  colloid (a), the diameter of blue laser in  $\text{AgNP}_s$  colloid (b), the diameter of blue laser in  $\text{AgNP}_s$  suspension(c), the diameter of green laser in  $\text{AgNP}_s$  suspension (d)

## 2.9 power meter:

The Photo detector (model UNO-200982, gentec-EO, Canada) is used to measure laser power. It working on the standard alkaline batteries. The figure (2.10) show the power meter



**Fig (2.10): detector and power meter**

The feature of the power meter are listed in the table (2.3)

**Table (2.3): the feature of the power meter [123]**

Power Range	5pW to 10kW
Monitor Accuracy	1%
Digital Size	76x57mm
Display	LCD
Dimensions	210Wx122Hx44D mm
Detector Types	Thermopiles, Photo Detector(PH Series)

## **2.10 CCD camera:**

A CCD camera (model Beamage - CCD12, gentec-EO, Canada) used to display nonlinear diffraction patterns. Figure (2.11), while the figure (2.12) show the picture of program of ccd camera



Fig (2.11):ccd camera

The most important features of CCD Camera are mentioned in the table (2. 4).

Table (2.4): The most important features of Camera ccd[ 124]

Wavelength Ranges	350-1150 nm
Sensor Technology	CCD
Damage Thresholds	10 W
Effective Aperture	6.3x4.8mm
Shutter Type	Synchronous
Pixel Dimension	4.65 x 4.65 mm
Pixel Count	1.4 M Pixels
Dimensions	61H x 81.1W x 22.9D mm

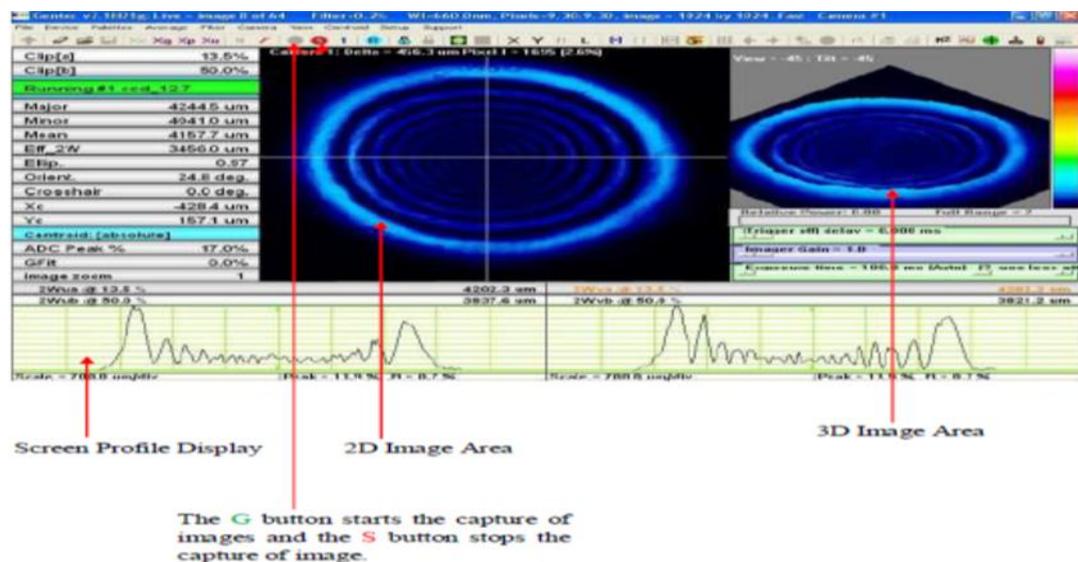


Figure (2.12): the trace of CCD camera

## 2.11 Attenuator:

The laser power is controlled by attenuator as shown in fig (2.13).  
(edmund industrial optics barrington, nj. Polarizing 24 mm japan)



Figure (2.13): Attenuator

## 2.12 Laser Source at 532 nm cw laser (green laser):

This laser emits a wavelength at 532nm (cw laser) (model DPSSL-MGL-III-532nm, CNI, China) this laser is pumped by the diode laser as shown in figure (2.14)

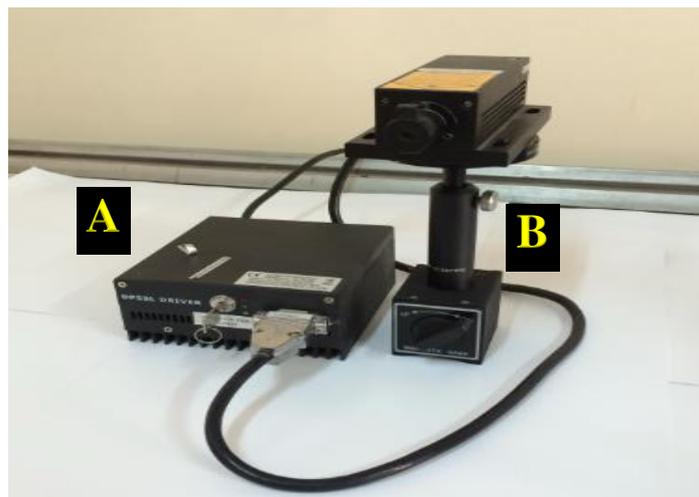


Figure (2.14): A: Power Supply, B: laser source

The following table (2.5) shows the most important qualities of this laser

**Table (2.5): Specifications of the green laser [125]**

Max. Output Power	208mW
Power Stability over 4 hours	4 hours <10%
Operating Mode	CW
Transverse Mode	TEM <sub>00</sub>
Beam Diameter	0.946mm

### **2.13 descriptions of chemical and physical properties of AgNP<sub>s</sub> suspension and colloid:**

The properties of silver nanoparticles are very important to know and control the ways in which nanomaterial's are manufactured and applied. There are several techniques that used to know these qualities:

#### **UV–Vis spectroscopy, FTIR, SEM, part per million (ppm) and TEM**

##### **2.13.1 UV-VIS Spectrophotometer:**

Spectroscopy (uv\_vis) was used to confirm the formation of nano silver particles. The absorption spectrum of the sample was measured using (Shimadzu UV-VIS 1800 spectrophotometer) covering the wavelengths (190\_1100) nm as shown in the figure (2.15).The examination was conducted at University of Baghdad College of Science Department of Chemistry Sciences



**Figure (2.15): The (uv\_vis) spectrophotometer**

### **2.13.2 Transmission electron microscopy (TEM):**

The electron microscope is used to identify the size, shape and form of nanoparticles. As shown in the figure (2.16). Where a model (Philips CM 10) with a 80 kV and 60 kV acceleration was used. The examination was conducted at the University Of Nahrain College Of Medicine



**Fig (2.16): Transmission Electron Microscopy (TEM)**

### **2.13.3 Fourier Transformation Infrared Spectrometer (FTIR):**

FTIR was used to identify molecular structure of AgNP<sub>s</sub>. The Fourier infrared spectra were obtained using (shimadzu 8300/8700). As shown in Fig (2.17). (FTIR) or called Fourier Transformation Infrared Spectrometer is a technique that used to resolve the chemical texture of many material such fibers, bulk , liquids, thin films, , powders solids, pastes, and else forms .Thus, it is used for a qualitative analysis of materials.The examination was conducted at University of Baghdad College of Science Department of Chemistry Sciences



**Fig (2.17): Fourier Transformation Infrared Spectrometer (FTIR).**

### **2.13.4 Scanning Electron Microscopy (SEM):**

A scanning electron microscope (Tesscan Mira3, France.2018) is a type of electron microscope that produces surface images of the terrain of samples to be examined by shedding a beam of electrons. They interact with sample atoms and produce images. This examination is used to study the topography and size of the prepared particles Figure (2.18) show the SEM microscopy. The examination was conducted at Iran



**Figure (2.18): SEM microscopy**

### **2.13.5 Part per million (ppm):**

(SHIMADZU AA-7000-The flame model) also called Atomic absorption spectrometer. It is used to measure the concentration of silver nano particles in units part per million (ppm), as shown in the figure (2.19). The examination was conducted at the University of Baghdad College of Education Ibn al-Haytham



**Figure (2.19): part per million measurements**

# Chapter Three

## *Results and Discussion*

### 3.1 Introduction:

The optical limit was investigated in (AgNPs) colloid and (AgNPs) suspension by using two types of lasers: blue laser at wavelength laser (473 nm) and green laser at (532nm) wavelength also study the effect of the power and wavelength of lasers on the formation of non-linear diffraction rings with graphed the relation between the power density of laser and number of rings, in addition non-linear refractive index and maximum change in nonlinear diffraction were calculated. Further more physical and chemical properties of silver colloid and silver suspension Nanoparticle were studied

### 3.2 Characteristics of AgNPs colloid:

#### 3.2.1 TEM measurement:

Transmission electron microscope was used to determine the particle size .Where the assay showed that the particle size formed was (15-30) nm and have spherical shape as shown in figure (3.1)

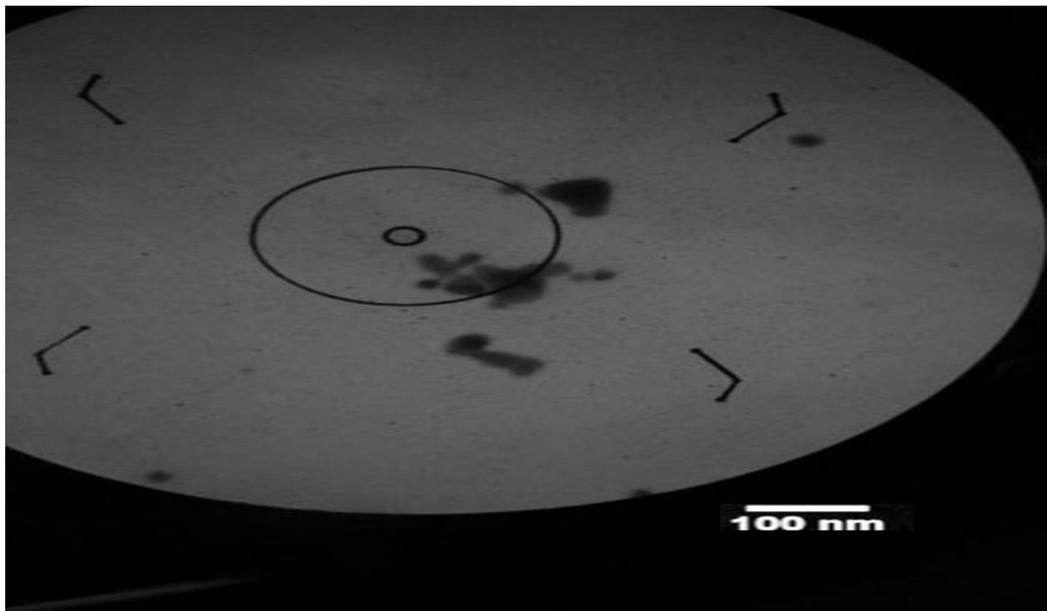


Figure (3.1 ): TEM measurement of AgNPs colloid

### 3.2.2 The ( uv\_vis) spectrometer measurement:

The ( uv\_vis) spectrum showed absorption of silver nanoparticles at the spectrum range between ultraviolet and visible rays due to (SPR)surface Plasmon resonance and that prove formation AgNP<sub>s</sub>[126] . The position of peak (SPR) depends on (size and shape of particle etc.) The peak of absorption at Peak", 393 nm" proves creation particle size in near 15 nm this result is identical to previous studies [127]. Also appear peak in rang (350\_600) nm prove formation AgNP<sub>s</sub> colloid [128][129].as shown in figure(3.2)

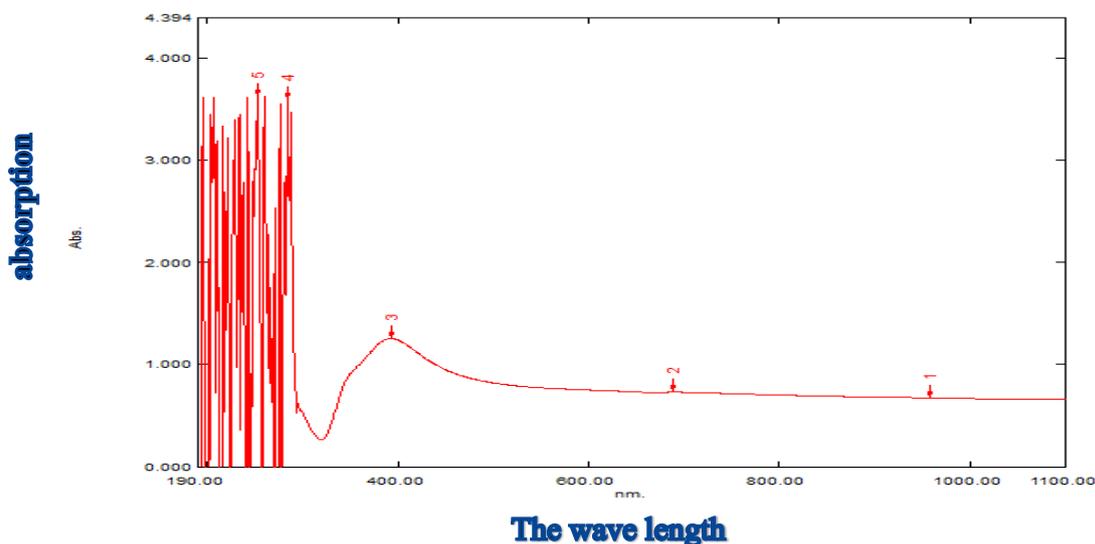


Figure (3.2): (uv\_vis) spectrometer measurement of AgNPs colloid

### 3.2.3 FTIR measurement:

Two bands were appeared at  $700\text{ cm}^{-1}$  and  $750\text{ cm}^{-1}$  two peaks correspond to metal–oxygen (M–O) bond due to (C–O<sup>-</sup> Ag<sup>+</sup>) bonds. There was also new band at  $1234\text{ cm}^{-1}$  due to the (C–NO<sub>3</sub>) bonds. The absorption bonds of (C–H) band were appeared at  $1375\text{ cm}^{-1}$  and  $1450\text{ cm}^{-1}$ . The (C–H) aliphatic absorption band was appeared at  $2990\text{ cm}^{-1}$ , it was observed shifts in wavelength added to some peaks, this proves present silver particles as a dispersed substance inside the prepared solution as showed in figure (3.3)

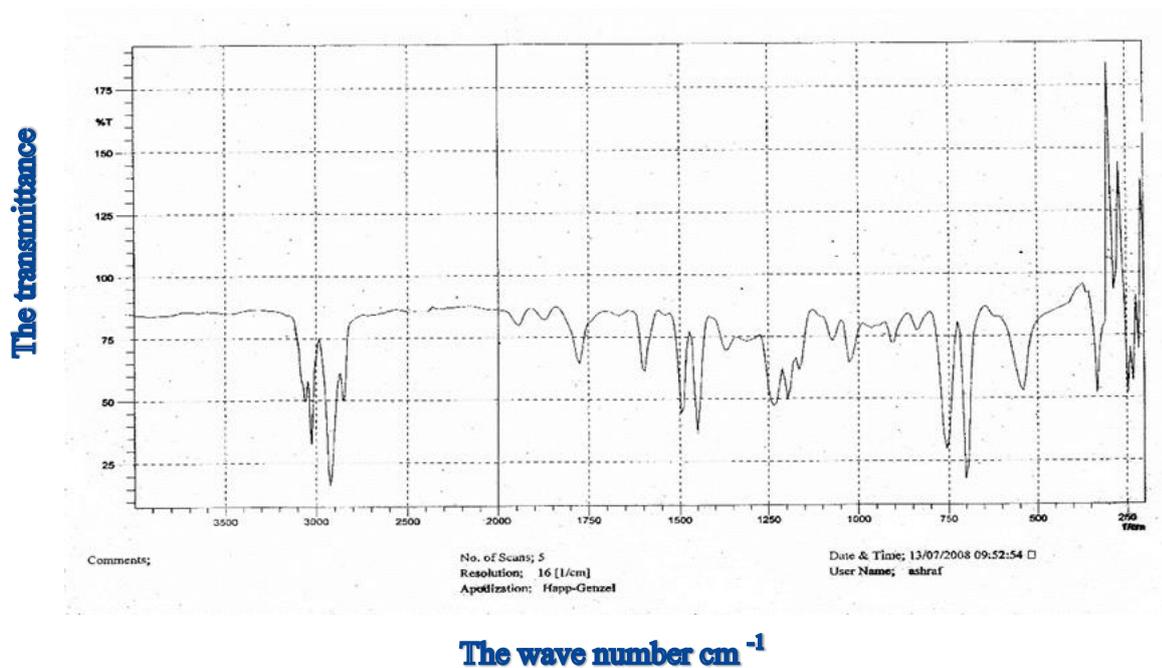


Figure (3.3): FTIR measurement of AgNPs colloid

### 3.2.4 Part per million (ppm):

This test was used to determine the amount of silver nanomaterial dissolved in deionized water. The test showed that sample (a) contained 31 ppm and sample (b) contained 11 ppm as shown in figure (3.4)

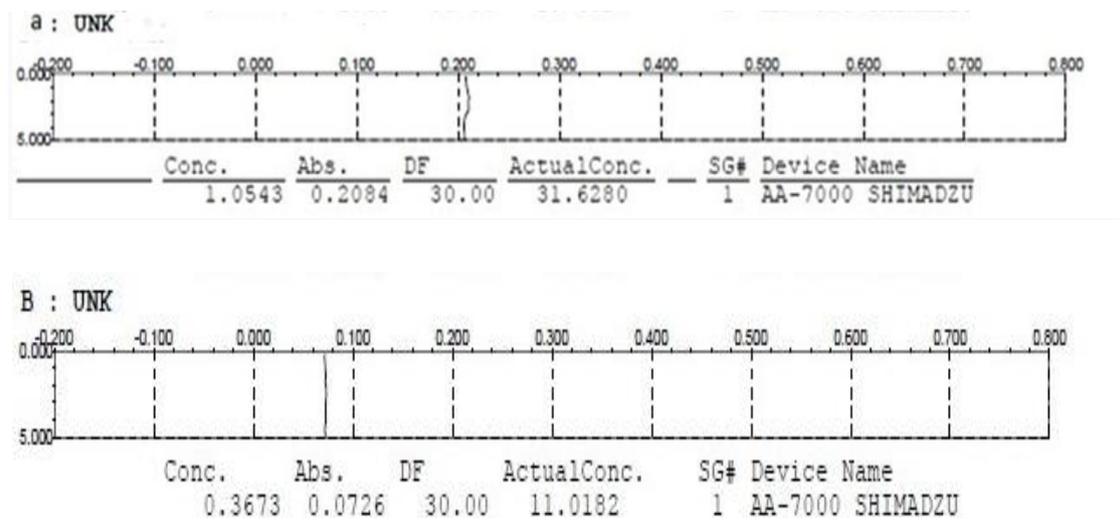
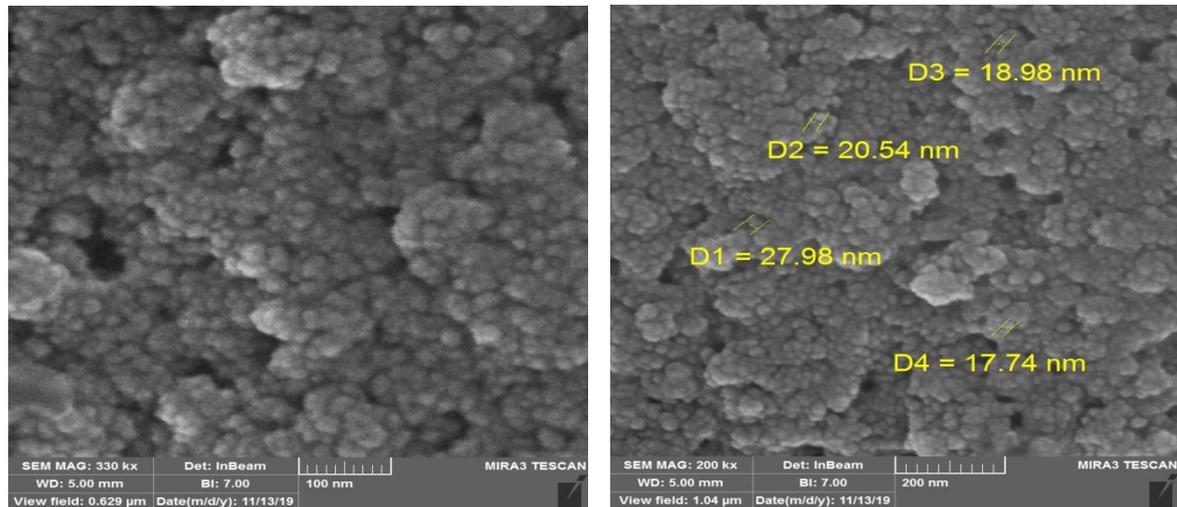


Figure (3.4): show the part per million of AgNPs colloid

### 3.2.5 SEM measurement:

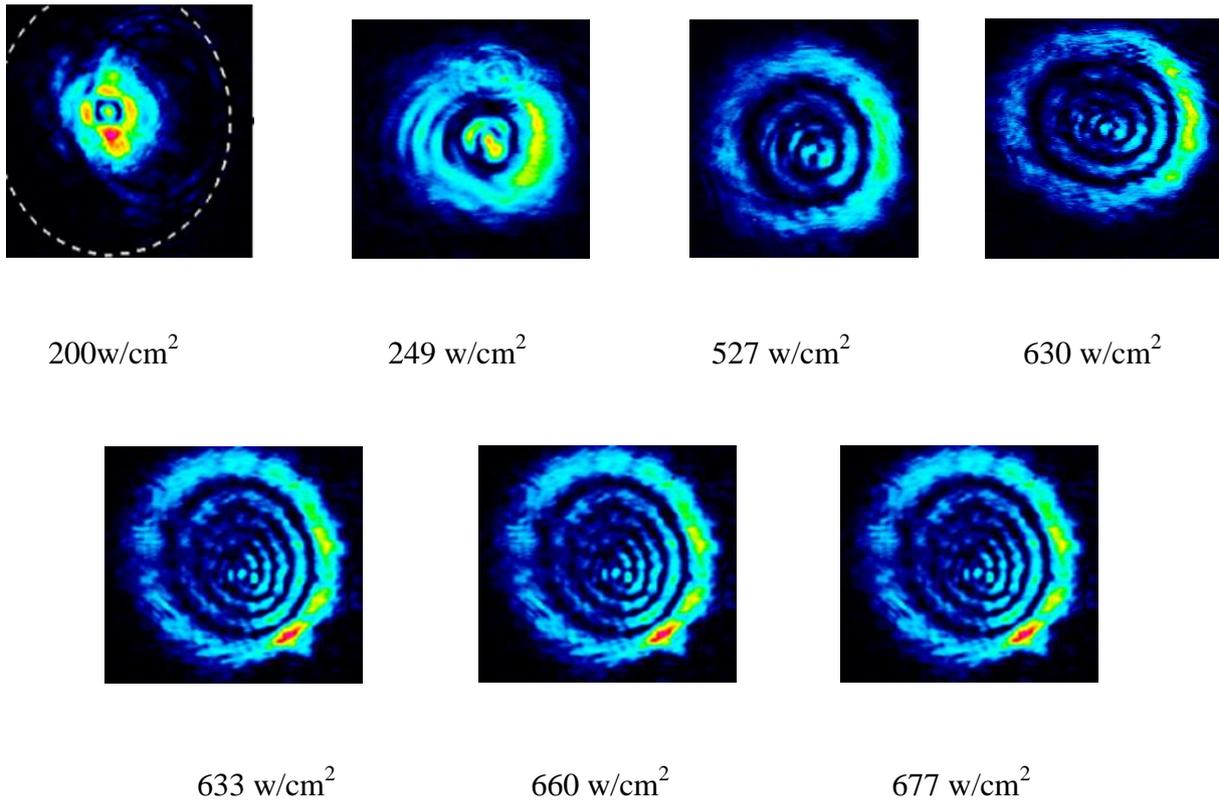
The particle size and topography of the silver colloid particles were determined by the examination of the images showed a particle size ranging from (17\_27) nm with spherical approximations as show in figure (3.5)



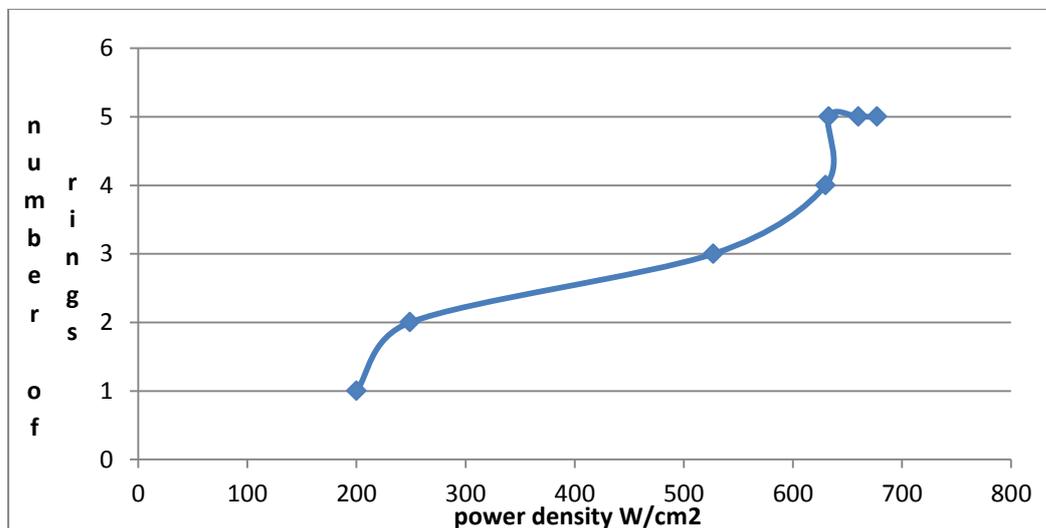
**Figure (3.5): SEM measurement of AgNps colloid**

### 3.3 Effect blue laser (473nm) on the AgNps colloid:

In sample a (31ppm) : the non-linearity phenomenon occur when creation The first non-linear diffraction ring at laser intensity ( $200\text{W}/\text{cm}^2$ ) the number of rings continued to increase gradually with the laser intensity increasing until the number of rings reached 5 at the laser intensity ( $633\text{W}/\text{cm}^2$ ). The increase in the intensity of the fallen incident laser from the range (  $633\_677$  )  $\text{W}/\text{cm}^2$  did not cause any increase in the number of rings due to the occurrence of an optical limited effect as shown in the figure (3. 6)and Graph the relationship between the laser intensity with the number of diffraction rings as in the figure(3.7)

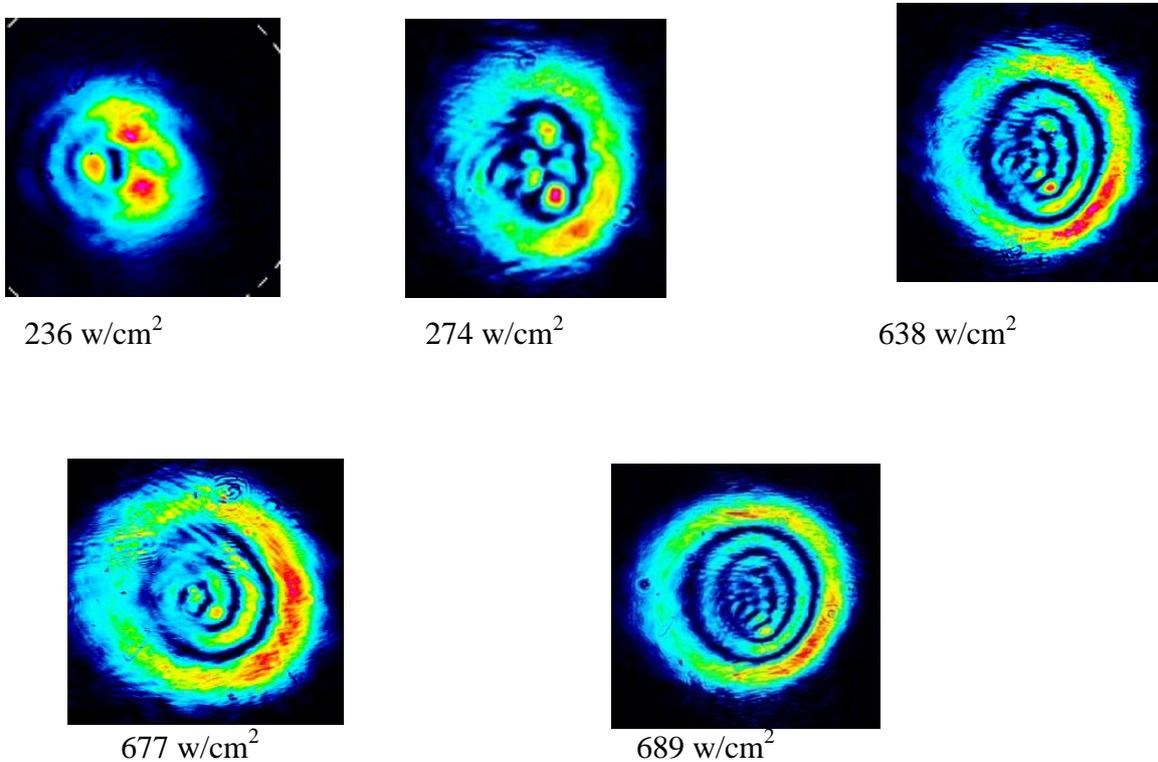


**Figure (3.6):** The non-linear diffraction rings sample (a) that have concentration 31ppm at different power density at range (200 \_677 ) W/cm<sup>2</sup>

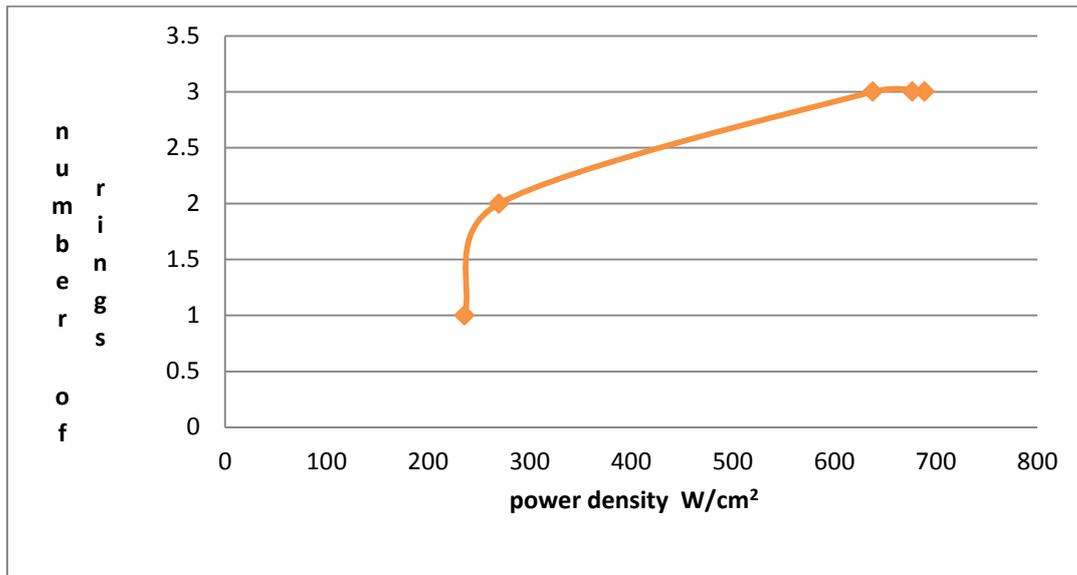


**Figure (3.7):** The relationship between the number of nonlinear diffraction rings and the laser intensity in sample (a)

In sample b (11ppm): : the non-linearity phenomenon occur when creation the first non-linear diffraction ring at laser intensity ( $236\text{W}/\text{cm}^2$ ) and the number of rings continued to increase gradually with the laser intensity increasing until the number of rings reached (3) at the laser intensity ( $638\text{W}/\text{cm}^2$ ). The increase in the intensity of the fallen laser from the range ( $638\_689$ )  $\text{W}/\text{cm}^2$  did not cause any increase in the number of rings due to the occurrence of an optical limited effect as shown the figure (3.8) and Graph the relationship between the laser intensity with the number of diffraction rings as shown in the figure(3.9).



**Figure (3.8):** The non-linear diffraction rings of samples (b) that have concentration 11 ppm at different power densities at range ( $236\text{-}689$ )  $\text{W}/\text{cm}^2$



**Figure (3.9):** The relationship between the number of nonlinear diffraction rings and the laser intensity in sample (b)

### 3.3.1 Determination of maximum change of nonlinear refractive index and nonlinear refractive index $N_2$ of AgNPs

The nonlinear refractive index and maximum change of nonlinear refractive index of AgNPs colloid at different concentrations was calculated by equations

$$\Delta n_{nl,max} = (\lambda_{beam} / L_{material}) * N_{rings} \quad (3.1)$$

Where  $\Delta n_{nl,max}$  refer to maximum change of nonlinear refractive index,  $\lambda$  refer to wavelength of laser 473 nm ,L refer to thickness of material 5mm

$$N_2 = \Delta n_{nl,max} / I \quad (3.2)$$

Where  $N_2$  refer to the nonlinear refractive index,  $I$  refer power density of laser. Tables (3.1)(3.2) showed the relation between nonlinear refractive index  $N_2$  and maximum change of nonlinear refractive index in AgNPs colloid in sample a (31)ppm and sample b(11)respectively ,the result showed the value of both the  $N_2$  and  $\Delta n_{nl,max}$  proportional with power density of laser Where the value( $\Delta n_{nl,max}$ ) increases with increasing intensity of the laser and become constant after optical limitation effect ,

which indicates that the relationship between them is direct. While the value of non-linear refractive index decreases with increasing laser intensity, which indicates that the relationship between them is inverse

**Table (3.1): The relation between nonlinear refractive index N2 and ( $\Delta_{nl,max}$ ) for AgNPs sample a (31)ppm as function of power density**

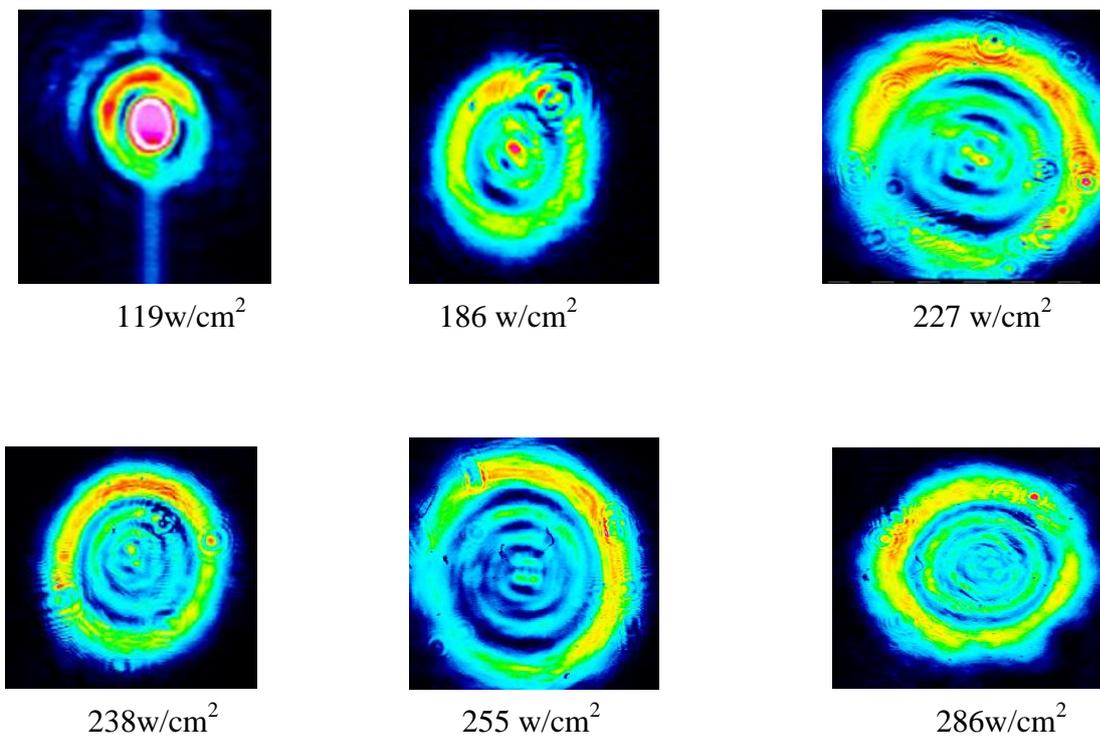
Number of rings	Power Density w/cm <sup>2</sup>	$\Delta_{nl,max} * 10^{-6}$	$N2 = (\Delta_{nl,max} / \text{Power density}) (w / cm)^{-1} * 10^{-6}$
5	677	4730	6.98
5	660	430	7.16
5	633	4730	7.47
4	630	378.4	7.50
3	527	283.8	8.97
2	249	189.2	18.99
1	200	94.6	23.65

**Table (3.2): The relation between nonlinear refractive index N2 and ( $\Delta_{nl,max}$ ) for AgNPs sample b (11) ppm as function of power density**

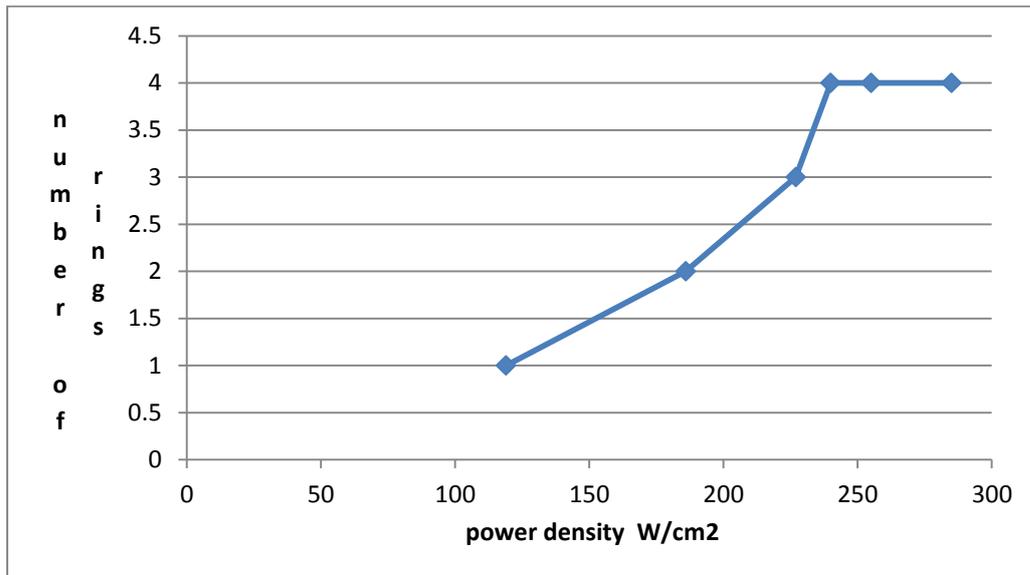
Number of rings	Power density w/cm <sup>2</sup>	$\Delta_{nl,max} * 10^{-6}$	$N2 = (\Delta_{nl,max} / \text{Power density}) (w / cm)^{-1} * 10^{-6}$
3	689	283.8	0.411
3	677	283.8	0.419
3	638	283.8	0.444
2	274	189.2	1.035
1	236	94.6	1.203

### 3. 4 Effect green laser (532nm) on the AgNps colloid:

In sample a ( 31ppm) : the non-linearity phenomenon occur when creation the first non-linear diffraction ring at laser intensity ( $119 \text{ W/cm}^2$ ) and the number of rings continued to increase gradually with the laser intensity increasing until the number of rings reached 4 at the laser intensity ( $238 \text{ W/cm}^2$ ). The increase in the intensity of the fallen incident laser from the range ( $238 \text{ - } 286$ )  $\text{W/cm}^2$  did not cause any increase in the number of rings due to the occurrence of an optical limited effect the figure (3.10) and Graph the relationship between the laser intensity with the number of diffraction rings as in the figure(3.11)

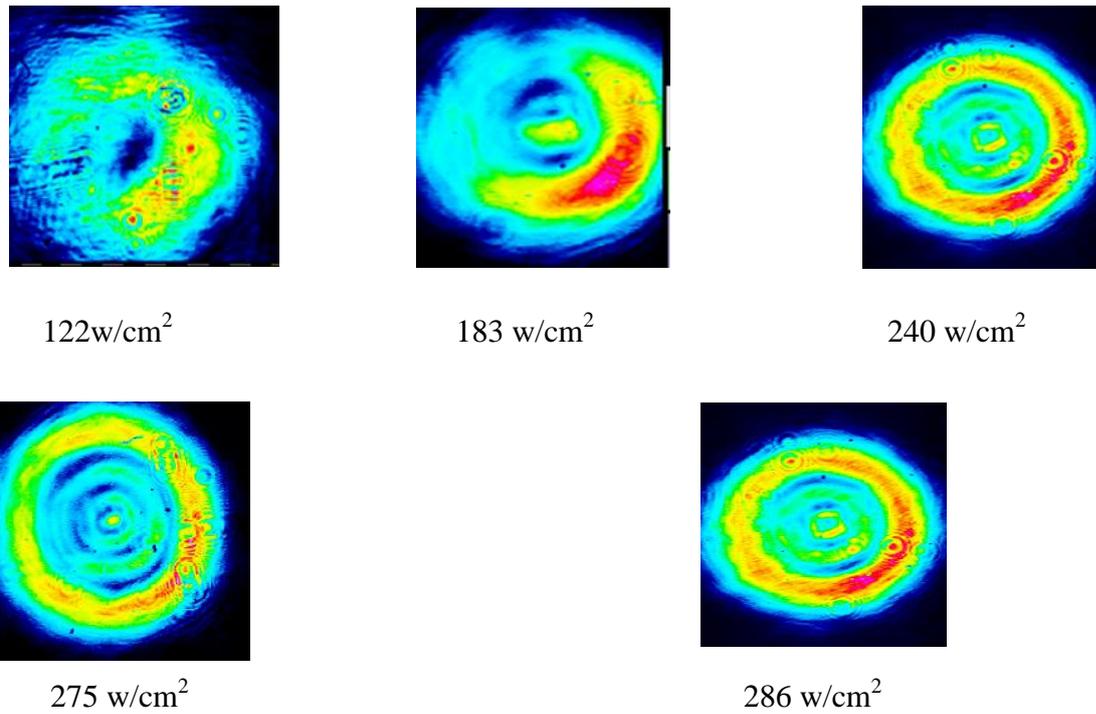


**Figure (3.10):** The non-linear diffraction rings of ( a) sample that have concentration 31ppm at different power density at range ( $119 \text{ - } 286$ )  $\text{W/cm}^2$

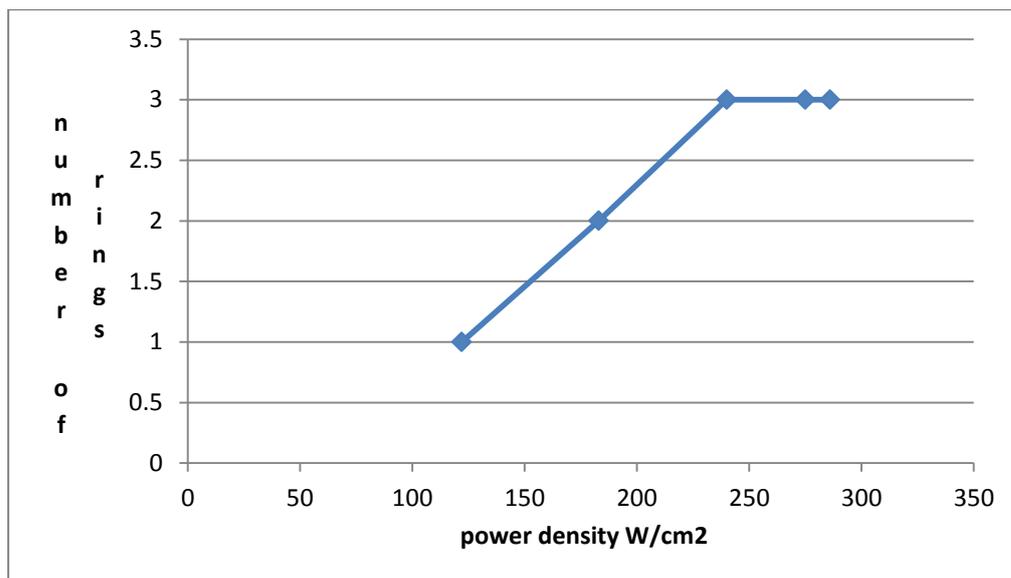


**Figure (3.11): illustrate the relationship between the number of nonlinear diffraction rings and the laser intensity in (a) sample**

**In sample b(11)ppm:** the non-linearity phenomenon occur when creation the first non-linear diffraction ring at laser intensity ( $122 \text{ W/cm}^2$ ) and the number of rings continued to increase gradually with the laser intensity increasing until the number of rings reached 3 at the laser intensity ( $240 \text{ W/cm}^2$ ). The increase in the intensity of the fallen incident laser from the range ( $240 \text{ - } 286$ )  $\text{W/cm}^2$  did not cause any increase in the number of rings due to the occurrence of an optical limited effect as shown the figure (3.12) and Graph the relationship between the laser intensity with the number of diffraction rings as shown in figure (3.13)



**Figure (3.12):**The non-linear diffraction rings of sample (b) that have concentration 11 ppm at different power density at rang (122 \_286) W/cm<sup>2</sup>



**Figure (3.13):** The relationship between the number of nonlinear diffraction rings and the laser intensity at sample (b)

### **3.4.1 Determination of maximum change of nonlinear refractive index and nonlinear refractive index $N_2$ of AgNPs**

The nonlinear refractive index and maximum change of nonlinear refractive index of AgNPs colloid at different concentrations was calculated by using equations (3.1) (3.2)

Tables (3.3)(3.4) showed the relation between nonlinear refractive index  $N_2$  and maximum change of nonlinear refractive index in AgNPs colloid in sample a (31)ppm and sample b(11) receptively, the result showed the value of both the  $N_2$  and ( $\Delta_{nl,max}$ ) proportional with power density of laser Where the value( $\Delta_{nl,max}$ ) increases with increasing intensity of the laser and become constant after optical limitation effect, which indicates that the relationship between them is direct. While the value of non-linear refractive index decreases with increasing laser intensity, which indicates that the relationship between them is inverse

**The table (3.3): The relationship between nonlinear refractive index N2 and  $(\Delta_{nl,max})$  of sample a (31ppm) as a function power density**

Number of rings	Power density w/cm <sup>2</sup>	$(\Delta_{nl,max}) * 10^{-6}$	$N2 = (\Delta_{nl,max} / \text{Power density}) (w / cm)^{-1} * 10^{-6}$
4	286	425.6	1.488
4	255	425.6	1.669
4	238	425.6	1.788
3	227	319	1.874
2	186	212	2.288
1	119	106.4	3.576

**The table (3.4): The relationship between nonlinear refractive index N2 and  $(\Delta_{nl,max})$  of sample b (11ppm) as a function power density**

Number of rings	Power Density w/cm <sup>2</sup>	$(\Delta_{nl,max}) * 10^{-6}$	$N2 = (\Delta_{nl,max}) / \text{Power density} (w / cm)^{-1} * 10^{-6}$
3	286	319	1.11
3	275	319	1.16
3	240	319	1.32
2	183	212	1.74
1	122	106.4	2.61

results in these figures (3.6,3.8,3.10and 3.12) showed the occurrence of nonlinear diffraction in ( AgNP<sub>s</sub> )colloid when using a relatively high intensity the number diffraction rings increases with increasing laser intensity[ 110], until reach to the saturation (optical limiting effect) . It should be noted that the outer diffraction ring was thick compared to the inner rings and the number of rings grows with the input laser intensity It was also observed that non-linear diffraction rings are very clear and countable, indicating that colloidal silver is a homogeneous material without the need to move(stirring) before conducting the experiment[ 130] furthermore these figure smentioned previously indicate the presence of silver colloid nano particle material on the phenomenon of optical limit effect that's agree with previous studies [ 131].To explain how the phenomenon occurs ,when the laser intensity falls on a material that has nonlinear Characteristics and because of the use of the laser Gaussian beam type (TEM<sub>00</sub>) the phase distortion of the incident beam leads to a random rise in temperature within the material, causing a spatial distribution of the nonlinear refractive index and its proportionality to the intensity of the laser (Kerr effect). Due to the creation of nonlinear diffraction rings, by self-defocused technique that's prove the material has a negative refractive index[ 110] .When the fallen intensity increases, the absorption coefficient value of the material will increase also until the incident intensity reaches a limit known as the ( threshold intensity) at(633W/cm<sup>2</sup> at concentration 31ppm)and ( 638W/cm<sup>2</sup> at concentration 11ppm)when using the blue laser, while was(238W/cm<sup>2</sup>at concentration 31ppm)and ( 240 W/cm<sup>2</sup> at concentration 11ppm)when using the green laser .The absorption value is equal to the transmission intensity due to several processes occurring within the material such as dispersion ,collision and scattering. In other words, liquids such (silver colloid) containing high thermal expansion cause a

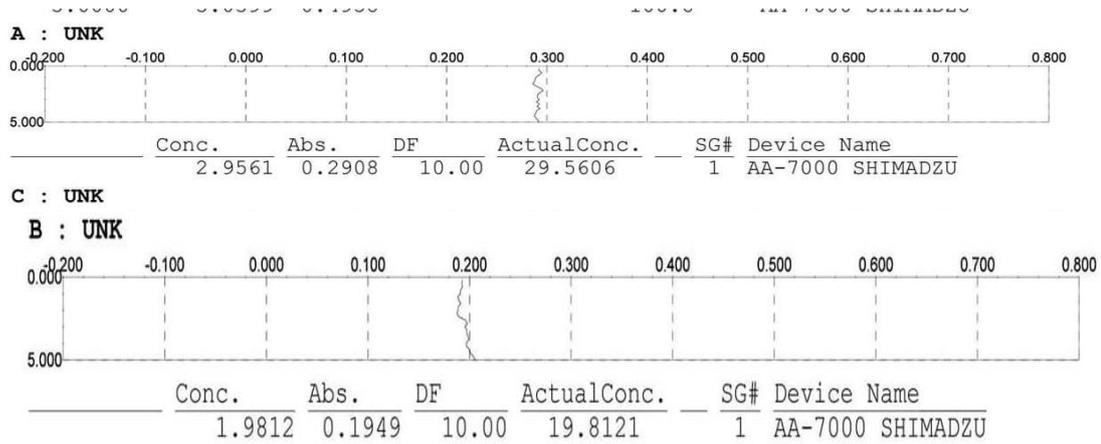
high absorption of the nonlinear material of the falling beam, which leads to high temperature and low density of the material, also heating by the laser is responsible for the change of the absorption coefficient value and the occurrence of optical limit [132] [133][134]. The results also showed that the lowest laser intensity for creation nonlinear diffraction phenomenon was (200\_236)  $\text{W/cm}^2$  at the blue laser and (119\_122)  $\text{W/cm}^2$  at the green laser. It was also observed that the threshold laser intensity to occur optical limiting in the blue laser was  $630\text{W/cm}^2$  at a concentration 31ppm, while the threshold intensity limit was 638 at the concentration 11ppm, and threshold laser intensity to occur optical limiting in the green laser was  $238\text{W/cm}^2$  at a concentration 31ppm, while the threshold intensity limit was  $240\text{ W/cm}^2$  at the concentration 11ppm which indicates that the materials with high concentration have a high optical limit. To illustrate the reason for this, the materials that have a high concentration (have a much number of particle per unit volume) and thus these particle will participate in nonlinear absorption processes in the material and these results are consistent with previous studies [135][136]. The result in table (3.1,3.2,3.3,3.4) referred value of maximum change of nonlinear refractive index and refractive index  $N_2$  of silver collide with power density of laser, the nonlinear refractive index decreased with increasing power density, induced the phase shift that be negative or positive corresponding the nonlinear material that occurred self-defocused and self-focused and result of formation central dark ring that prove the AgNPs is defocused material. This very corresponds to the reference [130]. It was also noted that the selection of the concentration of samples is very important in the nonlinear processes of the materials where when the concentration is high this leads to a bad result due to absorption and difficult transmission the laser beam and the sample with a low concentration does not play a role in nonlinear processes Thus the threshold limit of the concentration of nonlinear

material is very important in form nonlinear diffraction rings. As the high density of the outer rings compared with the inner rings indicate the occurrence of the nonlinear process of the third degree of colloidal silver as a result of the (**spm**) self-phase modulation. In addition, it was observed that the number of non-linear diffraction rings in 31ppm colloidal silver was 5 rings when using blue laser while 4 rings were used when green laser was used for the same concentration. The reason for this is due to the high energy of the photons (blue laser) compared with green the laser, which leads to an increase in the intensity of the laser falling on the material leading to high temperature of the material and increase the nonlinear interaction between the laser and the material. This is consistent with previous studies that demonstrate that the nonlinear properties of materials are strongly dependent on the laser wavelength as well as the pulse width [140]

### **3.5 Description of AgNPs suspension:**

#### **3.5.1 Part per million measurement:**

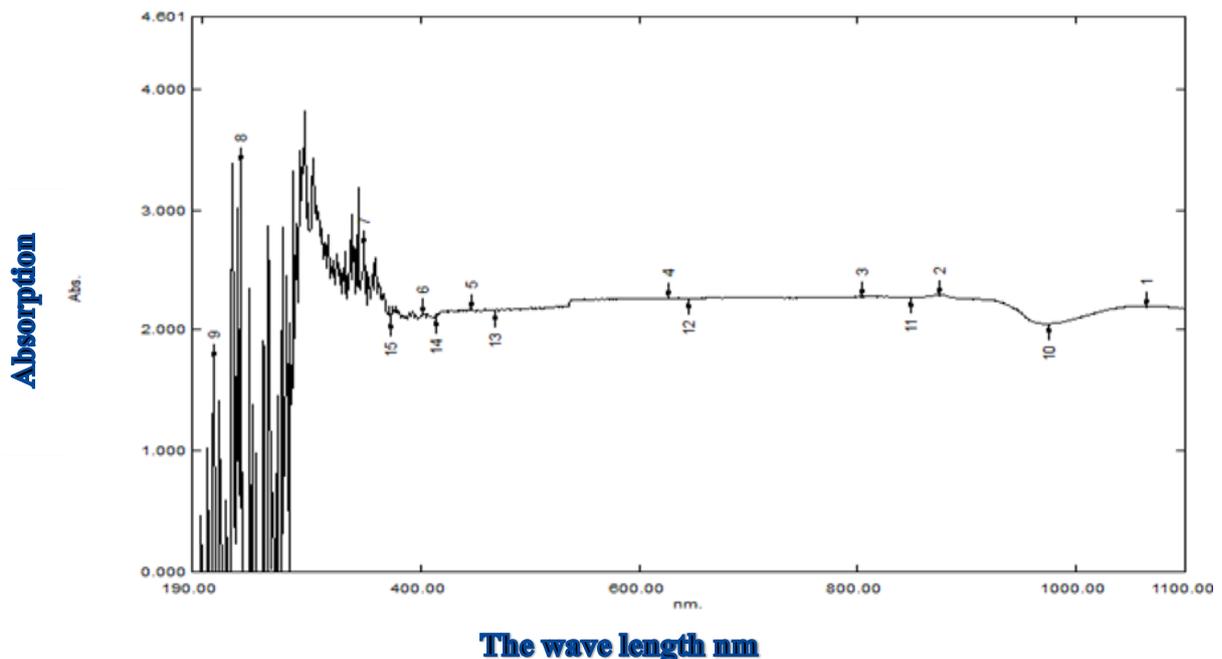
This test was used to determine the amount of silver nanomaterial dissolved in deionized water. The test showed that sample (A) contained 29 ppm and sample B contained 19 ppm as shown in figure (3.14)



**Figure (3.14) part per million measurements of AgNPs suspension**

### 3.5.2 The (uv-vis) measurement:

The (uv\_vis) spectrum showed absorption of silver nanoparticles at the spectrum range between ultraviolet and visible rays due to (SPR) surface Plasmon resonance and that prove formation AgNPs<sub>s</sub>[126]. The position of peak (SPR) depends on (size and shape of particle etc. ) The appearance of several absorption peaks at the range 400\_500 proves the formation of AgNPs as previous studies indicate [137][ 138]. The appearance of peaks at wavelengths above 500 (red shift) indicates nanoparticle aggregation as result left it for a long time



**Figure (3.15) (uv-vis) measurement of AgNPs suspension**

### 3.5.3 SEM measurement:

The examination of the images showed a particle size ranging from (27-44) nm with spherical approximations as show in figure (3.16)

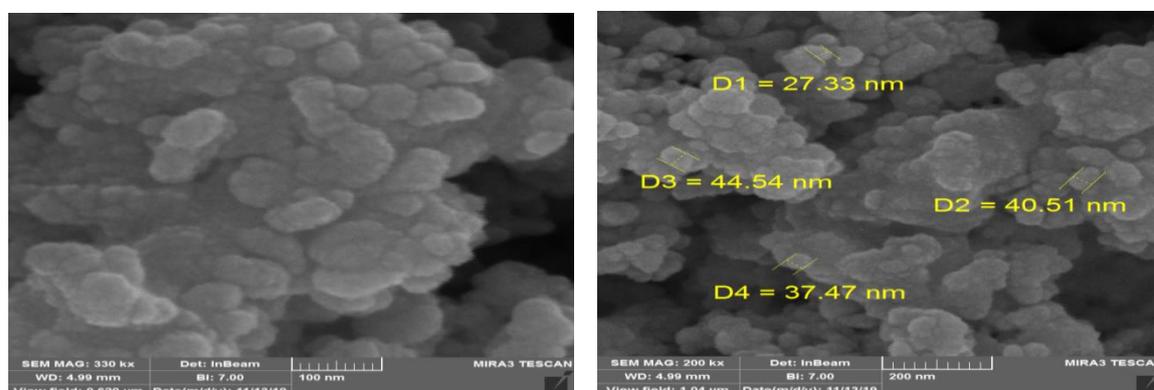


Figure (3.16): show the SEM measurement of AgNPs suspension

### 3.5.4 FTIR measurement

The FTIR spectrums discover a shift in the band values of the (NP), mostly from 400 to 3600  $\text{cm}^{-1}$ . This proves the presence of many Functional groups, also the(NP) bands were discovered at 400,600, 1100, 1400, 1750, 1920, 2300, and 2900  $\text{cm}^{-1}$  in the (FTIR)spectrum(Figure3.17).total, the presence of many functional groups signal that the synthesized( NP) has appeared in this solution.

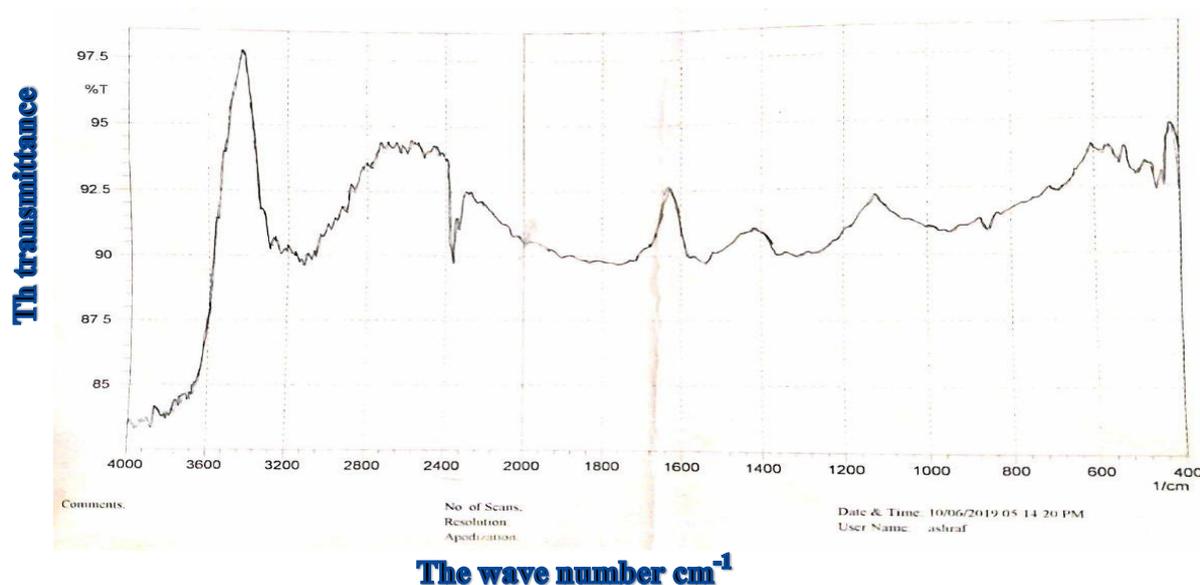
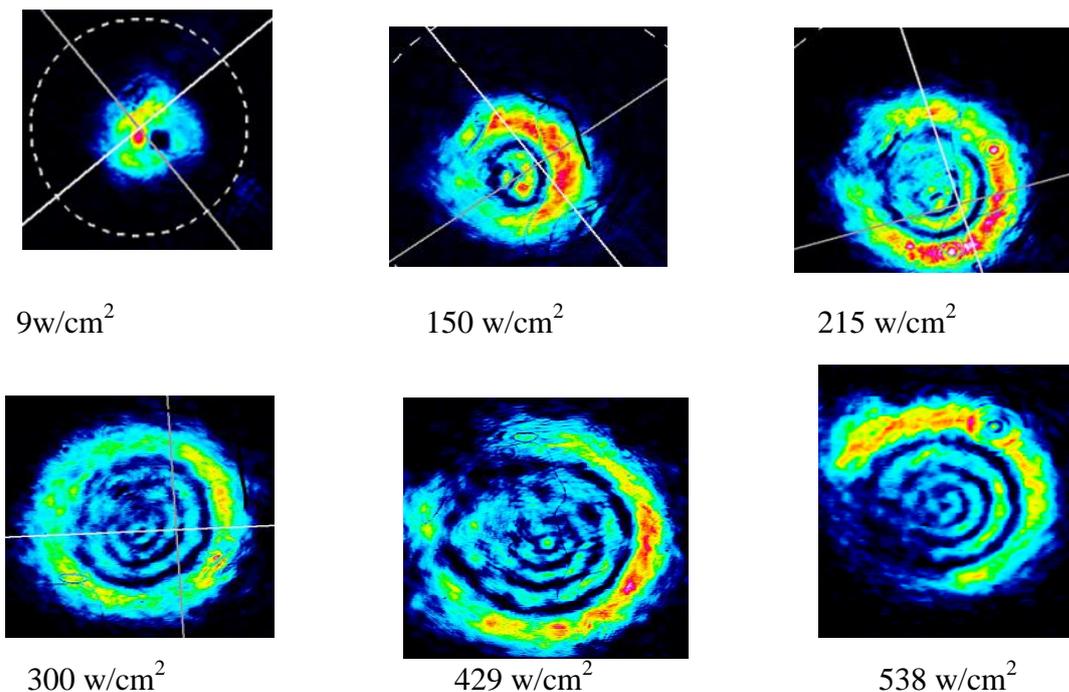


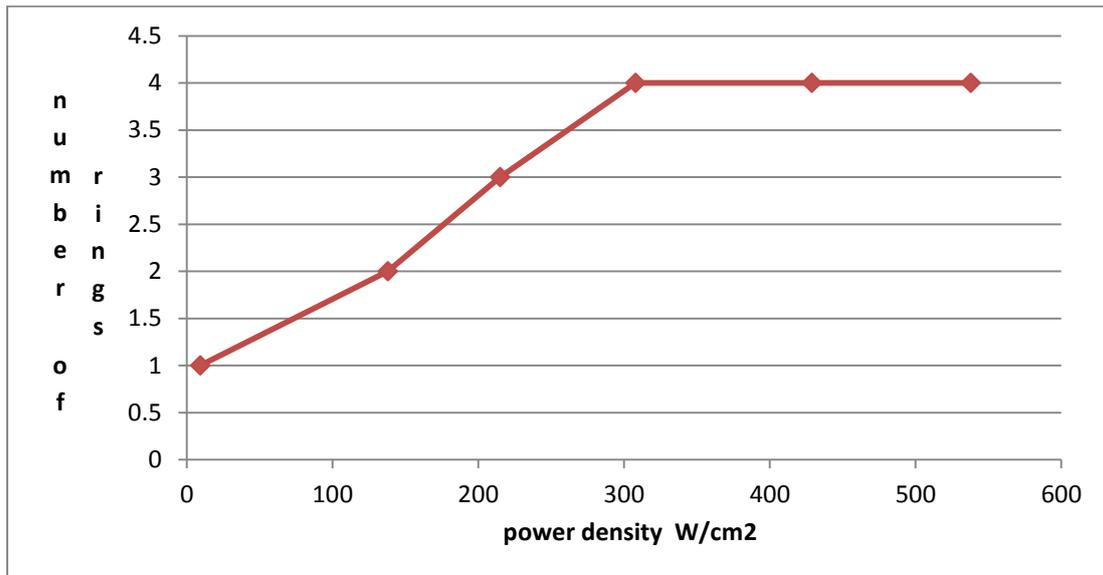
Figure (3.17): FTIR measurement of AgNps suspension

### 3.6 Effect blue laser (473nm) on the AgNps suspension:

In sample A (29ppm) 0.02 Wt. %: the non-linearity phenomenon occur when creation , the first non-linear diffraction ring at laser intensity( 9 W/cm<sup>2</sup>)and the number of rings continued to increase gradually with the laser intensity increasing until the number of rings reached 4 at the laser intensity(300 W/cm<sup>2</sup>) .The increase in the intensity of the fallen incident laser from the range (300 \_ 538 ) W/cm<sup>2</sup> did not cause any increase in the number of rings due to the occurrence of a optical limited effect as shown in the figure (3.18 )and Graph the relationship between the laser intensity with the number of diffraction rings as in the figure(3.19)

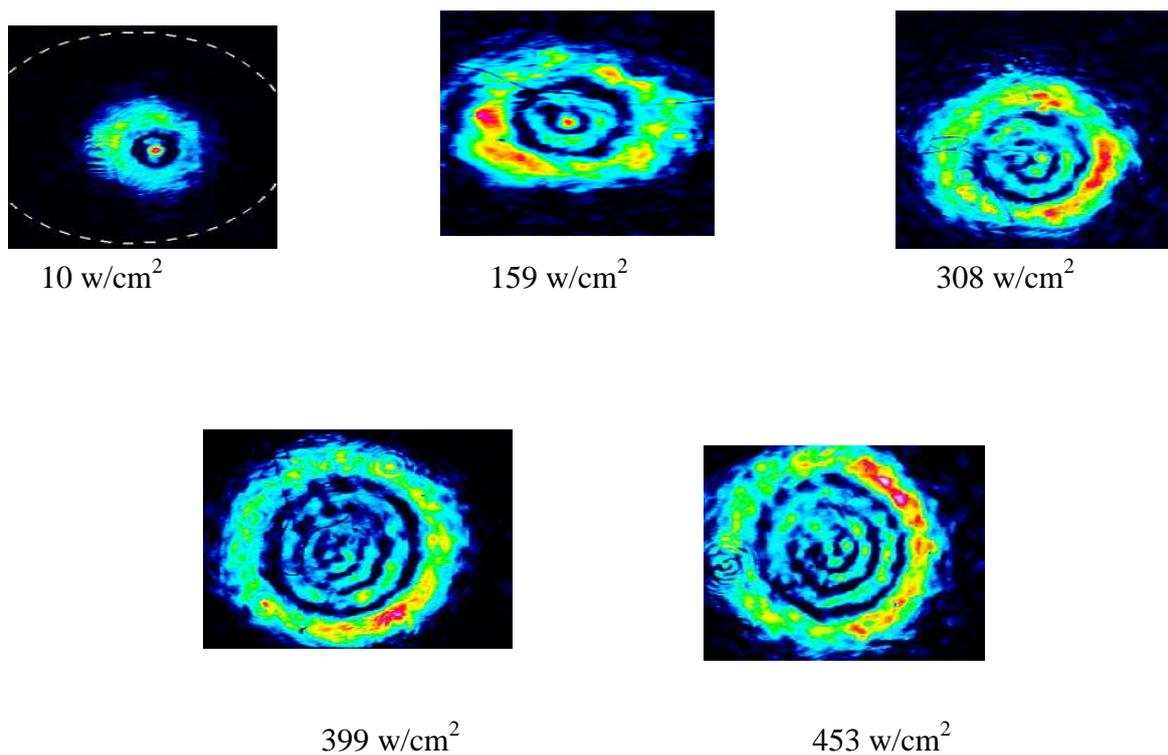


**Figure (3.18) :** The non-linear diffraction rings of sample(A) that have concentration 29ppm at different power density at range ( 9 \_ 538) W/cm<sup>2</sup>

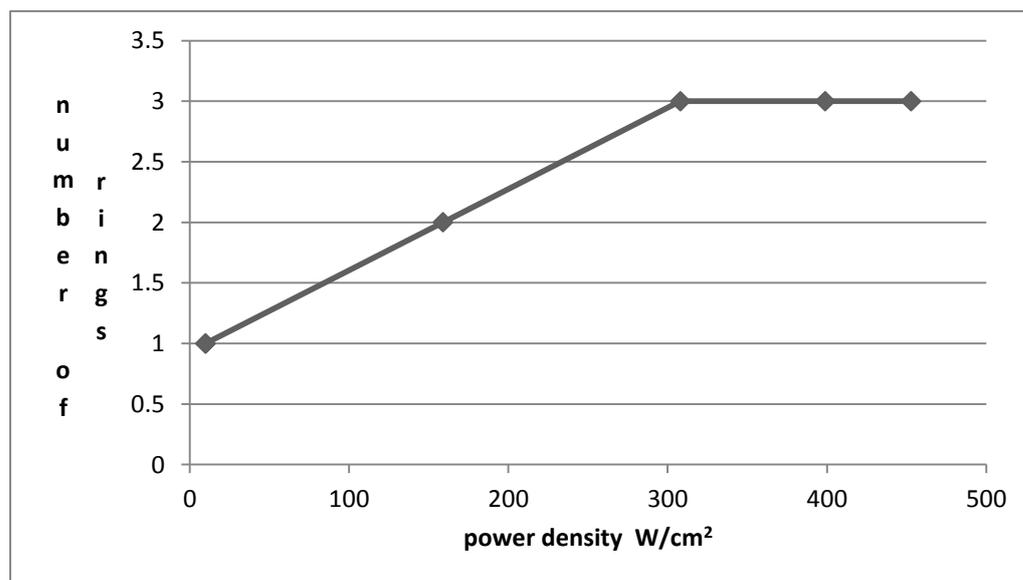


**Figure (3.19):** The relationship between the number of nonlinear diffraction rings and the laser intensity at sample (A)

In sample B( 19 ppm ) 0.009 Wt. % : the non-linearity phenomenon occur when creation the first non-linear diffraction ring at laser intensity ( $10 \text{ W/cm}^2$ ) and the number of rings continued to increase gradually with the laser intensity increasing until the number of rings reached 3 at the laser intensity ( $308 \text{ W/cm}^2$ ). The increase in the intensity of the fallen incident laser from the range (  $308 \text{ - } 453$  )  $\text{W/cm}^2$  did not cause any increase in the number of rings due to the occurrence of an optical limited effect the figure (3.20) and Graph the relationship between the laser intensity with the number of diffraction rings as in the figure(3.21).



**Figure (3.20):** The non-linear diffraction rings of sample (B) that have concentration 19 ppm at different power density at range (10 \_ 453) W/cm<sup>2</sup>



**Figure (3.21):** The relationship between the number of nonlinear diffraction rings and the laser intensity at sample (B)

### **3.6.1 Determination of maximum change of nonlinear refractive index and nonlinear refractive index $N_2$ of AgNPs**

The nonlinear refractive index and maximum change of nonlinear refractive index of (AgNPs) suspension at different concentrations was calculated by equations (3.1) and (3.2)

Tables (3.5)(3.6) showed the relation between nonlinear refractive index  $N_2$  and maximum change of nonlinear refractive index in AgNPs suspension sample A (29)ppm and sample B(19) respectively, the result showed the value of both the  $N_2$  and  $\Delta_{nl,max}$  proportional with power density of laser Where the value( $\Delta_{nl,max}$ ) increases with increasing intensity of the laser and become constant after optical limitation effect,, which indicates that the relationship between them is direct. While the value of non-linear refractive index decreases with increasing laser intensity, which indicates that the relationship between them is inverse

The table (3.5): The relationship between nonlinear refractive index  $N_2$  and  $(\Delta_{nl,max})$  for AgNPs suspension sample (A) (29)ppm as a function power density

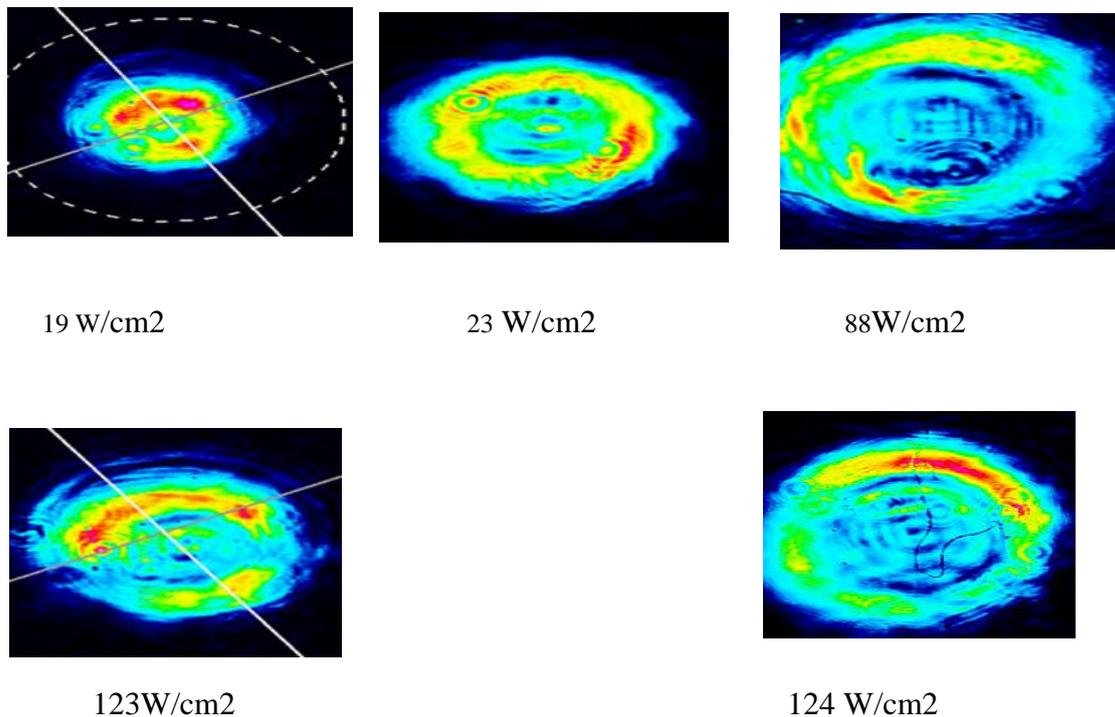
Number of rings	Power Density( w / cm <sup>2</sup> )	$\Delta_{nl,max} * 10^{-6}$	$N_2 = (\Delta_{nl,max} / \text{Power density}) (w / \text{cm})^{-1} * 10^{-6}$
4	538	378.4	0.70
4	429	378.4	0.88
4	300	378.4	1.26
3	215	283.8	1.76
2	150	189.2	2.52
1	9	94.6	42.04

The table (3.6): The relationship between nonlinear refractive index  $N_2$  and  $(\Delta_{nl,max})$  for AgNPs suspension sample (B) (19)ppm as function power density

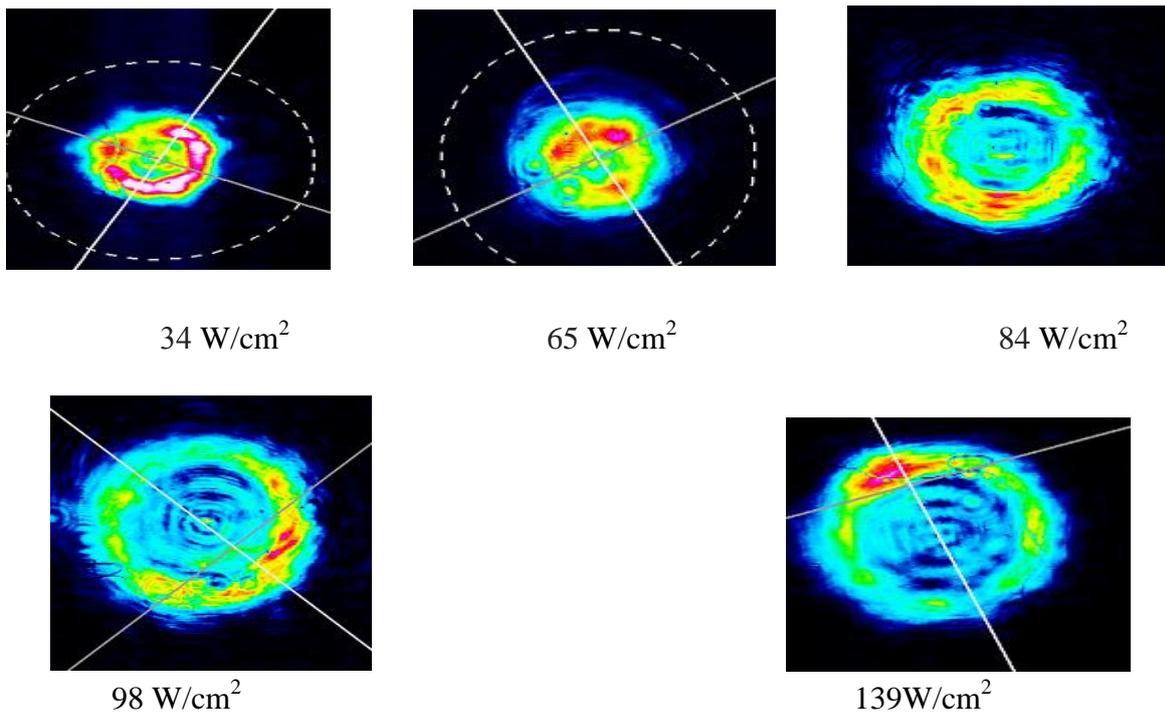
Number of rings	power Density(w /cm <sup>2</sup> )	$\Delta_{nl,max} * 10^{-6}$	$N_2 = \Delta_{nl,max} / \text{Power density} (w / \text{cm})^{-1} * 10^{-6}$
3	543	283.2	0.52
3	399	283.2	0.70
3	308	283.2	0.91
2	159	189.2	1.78
1	10	94.6	28.32

### 3.7 Effect green laser (532nm) on the AgNps suspension:

Blurred vision non-linear diffraction rings did not help to know the possession of silver suspension on the optical limiting or not as which could not calculate the number of rings of non-linear diffraction and determined the nonlinear refractive index and maximum change of nonlinear refractive index of silver suspension as showed figure (3.22and3.23) While the use of the same laser and the same material (silver suspension )with particle size (50nm) led to the formation of clear diffraction rings[115]. This shows that particle size plays a role in nonlinear optical processes and makes the nano fluid greet nonlinearity and this corresponds to previous studies [139]



**Figure (3.22):** The non-linear diffraction rings of sample (A) that have concentration (29) ppm at different power density at range (19\_ 124) W/cm<sup>2</sup>



**Figure (3.23):**The non-linear diffraction rings of sample (B) that have concentration 19 ppm at different power density at range (34\_ 139) W/cm<sup>2</sup>

The results in these figures (3.18 and 3.20) indicate the presence of silver suspension nanoparticle material on the phenomenon of optical limit effect when using (blue laser 473 nm) that's agree with previous studies [131], while the optical limit can't measure at the green laser result in Blurred vision non-linear diffraction rings as shown in figures (3.22 and 3.23). The results in these figures (3.18 and 3.20) showed occurrence of nonlinear diffraction in (AgNP<sub>5</sub>) suspension when using a relatively high intensity, the number diffraction rings increases with increasing laser intensity [110], until reach to the saturation (optical limiting effect). It should be noted that the outer diffraction ring was thick compared to the inner rings and the number of rings grows with the input laser intensity. It

was also observed that non-linear diffraction rings in suspended silver are less pronounced than colloidal silver when using the same laser (blue Laser) while diffraction rings were disarray when using green laser, the silver suspension material needs to be stirred by magnetic stirrer before use it in the experiment unlike the colloidal silver material. The results showed that the lowest laser intensity for creation nonlinear diffraction phenomenon was  $(10^{-9}) \text{ W/cm}^2$  at the blue laser and can't measure at green laser result in blurred vision non-linear diffraction rings. It was also observed that the threshold laser intensity to occur optical limiting in the blue laser was  $300 \text{ W/cm}^2$  at a concentration 29ppm, while the threshold intensity limit was 308 at the concentration 19 ppm, which indicates that the materials with high concentration have a high optical limit. To illustrate the reason for this, the materials that have a high concentration (have a number of particle per unit volume) and thus these particle will participate in nonlinear absorption processes in the material and these results are consistent with previous studies[ 135][136] .The result in table( 3.6and 3.7 ) referred value of and maximum change of nonlinear refractive index and nonlinear refractive index  $n_2$  of silver suspension with power density of laser , the nonlinear refractive index decreased with increasing power density, induced the phase shift that be negative or positive corresponding the nonlinear material that may be self-defocused and self-focused and result of formation central dark ring that prove the AgNPs is defocused material. This very corresponds to the reference [110]. It was noted that the selection of the concentration of samples is very important in the nonlinear processes of the materials where when the concentration is high this leads to a bad result due to high absorption and

difficult transmission the laser beam and the sample with a low concentration does not play a role in nonlinear processes. Thus the threshold limit of the concentration of nonlinear material is very important in form nonlinear diffraction rings. As the high density of the outer rings compared with the inner rings indicate the occurrence of the nonlinear process of the third degree of silver suspension as a result of the (spm) self-phase modulation. Experience also showed that the green laser 532 is not suitable for the study of the phenomenon of non-linear optical diffraction of silver suspension size (27\_44) nm. It was observed that the particle size plays an important role in determining the optical limit phenomenon of nonlinear nanoparticle as increasing the particle size increases the optical limit of the material (decrease of the threshold power density ) [141]. This case was observed when using a blue laser with a convergent concentration between colloidal silver (31ppm) and suspended silver (29ppm) where the first possessed a particle size (17-27)nm (threshold optical limit 630nm) while the other possessed a particle size ( 27-44) nm (threshold optical limit 300nm). It has also been observed that the concentration of samples (AgNP<sub>s</sub> colloid and suspension) increases the nonlinear processes of the material, leading to an increase in the number of nonlinear diffraction rings. This corresponds to previous studies [142]

### 3.8 Conclusion:

\* The number of non-linear diffraction rings for silver suspension and colloidal silver material increased with increasing laser power density

\*. The nonlinear refractive index  $n_2$  decreases gradually when the laser density is increased

\* Non Linear diffraction rings in colloidal silver (the base is di water) are more pronounced compared to silver suspension (the base is di water)

\*. Both silver collide and silver suspension has optical limiting effect

\* The green laser 532 is not suitable for the study of the phenomenon of non-linear optical diffraction of silver suspension size (27\_44) nm

\*The concentration of the material plays a role in the optical limiting effect nonlinear material

\*silver colloid doesn't required to stirring before experiment on the contrary silver suspended so as to contain the first anti-caking material

\*particle size plays a role in nonlinear optical processes and makes the nano fluid great nonlinearity

\*Increasing the size of a nanoparticle leads to an increase in the optical limit (low threshold intensity of the optical limit) for them so it is preferred to use in laser sensors

\*the value ( $\Delta_{nl,max}$ ) increases with increasing intensity of the laser and become constant after optical limitation effect, which indicates that the relationship between them is direct

\*Silver colloid is preferred because it is relatively cheap compared to silver suspensions and can be prepared in vitro and do not need to stir to contain material against caking

\*Material concentration plays a role in increasing the number of nonlinear diffraction rings in the nonlinear diffraction process

### **3.9 Future Works:**

1. Different thickness of cuvette can be used to study the nonlinear behavior of materials
2. The use of several nanoparticle fluids to study its nonlinear behavior
3. Several lasers with different wavelengths can be used to study the optical properties of nanomaterials
4. Comparison of colloidal silver (with acetone-based) with silver suspension (acetone-based) in terms of optical properties

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## الأهداء

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من اليقين أنّ شغفَ الإهداءِ أوّلُ الكرمِ وآخرُه.  
لأنّه يتوشحُ ببِلسمِ التّذكرِ والاعترافِ والشُّكرِ.  
ومَسارُ الاعترافِ يُشرقُ بضوءِ الشهداءِ أولاً.  
فاليكم أيُّها الاقربون روحاً وحبّاً ...

شهداء العراق الطاهرون، شهداء جيشنا الباسل، شهداء  
الحشد الشعبي المقدس وشهداء ثورة اكتوبر (المطعم التركي)  
جزءاً من طوقِ جميلكم الكبير.....

شهد العاني

## الخلاصة :

في هذه الرسالة تمت دراسة تأثير الحد البصري للمواد النانوية من خلال تقنية اللا تركيز الذاتي . تم استخدام نوعين من الليزر (الليزر الأزرق 373 نانومتر والليزر الأخضر 532 نانومتر) بالإضافة إلى استخدام نوعين من المواد النانوية (الفضة الغرواني) و(الفضة المعلق) لمعرفة تأثير الحد البصري بينهما، تم استخدام الماء الغير مؤين لكلا الحالتين كمحلول لهما . تم تصنيع تركيزات مخففة من فضة الغروانيه وهما (31 و 11) جزء في المليون وكذلك من الفضة المعلق وهما (29 و 19) جزء في المليون. ان دراسة سلوك الحد البصري لمادة فضة الغرواني يظهر امتلاكها للحد البصري عند استخدام الليزر الأزرق في كثافة الطاقة (633- 638) واط /سم<sup>2</sup> بتركيز (31 جزء في المليون و 11 جزء في المليون) على التوالي، وأيضاً بالليزر الأخضر بكثافة الطاقة (238\_240)-واط /سم<sup>2</sup> بتركيز (31 جزء في المليون و 11 جزء في المليون) على التوالي . ثانياً أظهر سلوك الفضة المعلق حدا بصريا عند استخدام الليزر الأزرق في كثافة الطاقة (300-308) واط/سم<sup>2</sup> بتركيز (29 جزء في المليون و 19 جزء في المليون) على التوالي ، في حين أن عدم وضوح حلقات الحيود غير الخطية كان في الليزر الأخضر، تم استخدام عدة اختبارات مثل فحوصات TEM و SEM و PPM و FTIR و (uv\_vis) علاوة على ذلك ، ثالثاً تم حساب مؤشر الانكسار اللاخطي والتغيير الأقصى في معامل الانكسار اللاخطية للتركيزات المخففة ورسمت العلاقة بين شدة الليزر المستخدم وعدد حلقات الحيود.



وزارة التعليم العالي والبحث العلمي

جامعة بغداد

معهد الليزر للدراسا العليا

دراسة ظاهرة الحد البصري والخصائص البصريه اللاخطيه  
للمواد الفضة النانوية الغروانية والمعلق بواسطه تقنيه اللاتركيز  
الذاتي

رسالة مقدمة الى

معهد الليزر للدراسات العليا/ جامعة بغداد

لإستكمال متطلبات نيل شهادة ماجستير علوم في الليزر/ الفيزياء

من قبل

**شهد مهند ناجي**

بكلوريوس علوم الفيزياء - 2011

بإشراف

**الاستاذ المساعد الدكتور محمد كريم ظاهر**

