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**Studies on synthesis and characteristics of metallic
nanoparticles**

A Thesis Presented

By

Xiao Li

To

The Graduate School

In Partial Fulfillment of the

Requirements

For the Degree of

Master of Science

In

Materials Science and Engineering

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Abstract of the Thesis

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The thesis mainly focused on the synthesis of nanoparticles by using sonolysis. Different precursors and solvents were used during the sonolysis. We analyzed the kinetics of sonolysis process to find the difference between the sonolysis using different precursors and solvents. The surface morphology and particle size were determined using SEM, TEM, and XRD. The data were then compared and analyzed.

Key words: sonolysis, nanoparticles, nanoparticle formation kinetics.

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Introduction

The first time humans made use of nanomaterials was over hundreds of years, but the concept of nanomaterials got mentioned just about 50 years ago [1]. Generally speaking, nanomaterials are those with a size between 1 nm to 100 nm [2]. Nanomaterials have found application in polymers, metals, ceramics, glasses and composites. At the nanoscale level, properties of nanomaterials are different compared to those of bulk materials. One of the most important differences is that the nanomaterials have increased surface area to volume ratio, which can lead to quite different properties from those of bulk.

The unique properties of nanomaterials make them have the potential to be widely used in chemical industry, medical use, automobiles and tiny electronic products area. For example, General Motors has produced the first car with nanomaterials used in exterior automotive applications. Also, more and more nanomaterial based catalysts are used because they have maximum surface area exposed to the reactant which leads to enhanced reaction rates. Nanotechnology has been improved to a point that academic researchers are interested because private companies invest a lot on the research in nanotechnology.

Nanotechnology is aimed to develop the devices, systems and structures with nanoscale materials. Under the research of decades, the nanotechnology has been improved from simple nanoparticles and powders to nano coats and layers, from optical and mechanical devices to medical devices. Today nanotechnology is still just at its beginning, but it is developing very fast. Currently, the technology focuses on minimizing the size, optimizing the structure and other properties of nanomaterials. In the future, the nanotechnology is anticipated to produce nanosystem, even molecular nanosystem that can make materials have molecular structures into direct use as devices.

Objective of This Research

The aim of the thesis was to compare the nanoparticles of iron and molybdenum, prepared under different conditions using a technique called sonolysis.

1 Review of Literature

1.1 Nanomaterials

The first time the concept of nanomaterials mentioned was in 1959 by physicist Richard Feynman [1]. After several decades of research, nanomaterials have been developed and applied in many different areas. More breakthroughs are expected in research of nanomaterials for applications in energy, health and materials in space areas.

Nanomaterials are usually defined as materials with small size in the range of 1 to 100 nanometers (nm) [2]. Their size are frequently compared with human hair which is tens of thousands times of one nanometer size. The components of nanomaterials range from metals, glasses, polymers to ceramics or composite materials, which also indicates the broad application of nanomaterials. Even though nanomaterials are the materials with mini size, nanotechnology is not as simple as just another step in miniaturization. In fact, nanomaterials substantially differ from bulk materials. For nanomaterials, what the most obvious difference with bulk materials is not just the size, but the properties. For example, the nano particles have larger surface area, there are more atoms available on the surface per unit volume. Due to the enormous unique properties and the bright potential applications of nanomaterials, the development of nanotechnology has been booming especially with the availability of more sophisticated scanning electron microscopy (SEM) and scanning tunneling microscopy (STM). During 1980s, a kind of hollow carbon spheres, called fullerenes, were discovered by researchers in Rice University that led to synthesis of carbon nanofibers [3]. A few years later, the researchers of NEC in Japan fabricated the first carbon nanotube which is now widely used in many areas [4]. Until 1999, the production of nanocomposites was up to a few millions pounds [4]. After ten years of development, the volume of nanomaterials used is exploding. However, a method that

can be used to produce nanomaterials in a narrow particle size range remains a challenge.

Nanoparticles are usually inorganic and made of metals, oxides, sulfides, or other forms. They are well known to have distinct mechanical, magnetic, electrical, chemical, optical and other properties. For example, copper nanoparticles with scale smaller than 50nm have better mechanic properties than bulk coppers [5]. They also have special optical properties as gold nanoparticles can be seen as red or even black in solutions. This is due to the quantum effects of nanoparticles. During the time nanomaterials were first developed, people's concerns about the influences of new materials on health and environment is growing. The influence of nanomaterials is due to their small size that allows them to easily absorb through the skin and during breathing or through swallowing. These materials have unknown effect on human bodies and this is a major area of research. To prove and solve this problem, several groups around the world, such as one from Sweden are leading research to find that copper oxide nanomaterials had the worst effect and it was seen as a health risk [6]. Besides copper, other metallic oxides nanoparticles were also researched but found that these only did little damage to DNA and were seen as harmless. Not only the health problem, the nanomaterials also have potential environmental problems, because their super tiny size, these nanoparticles are usually easy to get into natural system without any proper disposition.

1.2 Synthesis of Nanoparticles

In order to make use of nanomaterials in electronics, catalysis and medicine, the size, shape and crystalline structure of nanomaterials have to be strictly controlled. How to fabricate the nanomaterials with the best size, shape and crystalline structure is still a challenge. Hence a majority of scientists are finding better methods to synthesize nanoparticles. Synthesis of nanoparticles can be classified into two categories:

attrition and pyrolysis. The former is also called as 'top-down' while the latter as 'bottom-up' [7]. 'Top-down' involves division of macro or micro scale solid into smaller particles through milling or attrition. After the division, the particles are then air calcined to recover nanoparticles. 'Bottom-up' is a procedure to evaporate vaporous precursors under high pressure or high temperature, and then the particles left are air calcined to recover nanoparticles. The latter one is more popular because it's easier and time saving.

Nanoparticles have high surface area which increases free enthalpy of the dispersion system and make the process metastable. If the nanoparticle dispersion is not sufficient enough, the activation energy will not be not high enough and this would lead to aggregation of nanoparticles and the failure to form nano-sized particles. So it's very important to make sure the reaction conditions are favorable to achieve desired activation energy. However, the nanoparticle formations conditions cannot exceed some threshold because the nanoparticles are only kinetically stabilized. Some researchers use surface stabilization to stop the recrystallization and aggregation of nanoparticles during the synthesis procedure.

Thermal decomposition is usually used as a way to synthesize nanoparticles. Heat provides enough activation energy to form nanoparticles. In the last 20 years, an alternative method is under development--sonolysis. Sonolysis uses sound to provide enough energy to break chemical bonds in metal precursors and yield nanoparticles. In actual sonolysis method, sound waves cause acoustic cavitation [8]. With fixed frequency sound waves, the liquid will produce plenty of bubbles and the collapse of these bubbles in liquid can cause the cavities to form, grow and implode, which leads to high temperatures up to 5200 K and high pressures up to 30 MPa [9-10], creating conditions to break chemical bonds. The process then yields nanoparticles. In this paper, synthesis of metal nanoparticles was achieved by sonolysis of metallic carbonyl.

1.3 Application of Nanoparticles

As stated earlier, mainly due to higher surface area per unit volume, nanoparticles have different properties compared to their bulk. Nanoparticles have found applications in health, advanced electrical products, catalysts to produce fuels.

1.3.1 Application of Nanoparticles

Nowadays, researchers have developed many products with nanoparticles. Among all of these applications, the application in home cleaning maybe the most related to people's normal life. One kind of cleaning detergent which has nanoparticles inside has been produced, when the nanoparticles meet the dirt or grime, they can absorb them and make the place clean by itself. This kind of technology can also be used in some self-cleaning fabrics for the same reason.

The other important and also very promising application of nanoparticles is in medical area. Nanoparticles usually have the same size as pathogens, which could make them the best choice to fight against the viruses that are harmful to human bodies. When the nanoparticles are injected into bloodstream, they can fight with viruses just like the immune system do by itself. Similarly, because of the property of nanoparticles, they also have the potential to be used in curing AIDS patients by serving as immune system [11].

Besides the potential application as immune system, they are also hoped to be applied to fight against cancer. Currently the mostly used treatment for cancer patients is chemotherapy which could kill the cancer cells by transferring lethal chemical through the body to the location where the cancer cells are. However, using this treatment, because the chemicals are normally not in molecular size, it's almost

impossible to kill all of the cancer cells thoroughly. As we know cancer cells can reproduce very fast, thus this treatment is still not satisfied. Plus, at the same time that the lethal chemicals kill the cancer cells, they also could kill the healthy and useful cells. This disadvantage brings a great pain for the patients. Luckily, nanoparticles have the great potential to conquer all of these troubles. Firstly they have the molecular size and then have the ability to kill the cancer cells thoroughly. Secondly the nanoparticles can be equipped not only with drugs to kill cancer cells, but also the sensors even cameras to track these nanoparticles after they flow through the bloodstream. Using this way, the drugs can be shipped at the exact location and just kill the cancer cells while avoiding to kill the healthy ones.

Not only in the medical area, can nanoparticles be applied in many other fields such as optical. It has been hundreds of years that nanoparticles were added to produce stained glass windows. Nowadays nanoparticles have been used as a sensing approach due to their optical property. The nanoparticles can have different optical responses when there is a change in the nano environment, which can be transferred to signal transduction [12].

Nanoparticles have a tiny size which also makes them applicable in electronics, by adding nanoparticles can usually improve the performance of delicate electronics. The materials made of nanoparticles have conductivity with a high rate and can also be used in small consumer electronics like cell phones. For example, one of the most attractive parts of electronic products is the display screen, the digital displays made of nanoparticle electronics can have higher electricity efficiency, less cost to produce, and they can also have a bigger size, lighter weight and brighter color [13].

Nowadays, nanoparticles can be used in more and more fields, such as construction and aerospace. For most of mechanical devices, by adding nanoparticles in the device materials can usually improve the wear and tear resistance. Not only this, they can also provide anti-corrosion which is very important for materials used in

construction and aerospace. Because of the properties that nanoparticles have, new composites and structural materials with lighter weight and stronger mechanical performance can be made. Nanoparticles can also be used in batteries such as fuel cells, solar panels. They can provide higher energy density, better hydrogen storage, more efficient photoelectric conversion. Besides all of these applications, nanoparticles are also potential in other fields like biomedical, thermal, and magnetic area.

1.3.2 Application in Catalysts

More and more people have realized that the environmental problem is one of the most serious problems associated with nanoparticles. With the increasing world population, booming industry, and rapidly growing automobiles, fossil fuel reserves are falling and our environment is getting worse. Thus many researchers and governments have paid a lot of attention on solving this problem, such as reducing the harmful by-products and wastes during the manufacturing process to clean up the environment and prevent the pollution [14]. Also, the nanoparticle catalysts can be used in refinery industry. In order to improve the efficiency and reduce the cost of catalysis process, the catalysts particles should be made small and they can be dispersed so that the only desired reactions can be achieved [15]. Nanoparticles have extremely large surface to volume ratio that can help with catalytic reaction rates [15]. Nanoparticle catalysts have two roles on the catalytic reactions, firstly they can provide sites for catalysis, secondly they also be supported [16]. Nano particles can lower activation energy that can help with better product selectivity at lower temperatures [17]. Normally, the catalysts materials are made of metals, metal oxides, metal sulfides or metal carbides. By controlling the size, it can not only improve the performance, but also produce the desired products instead of wastes [18]. Besides the size control, the composition of the nanoparticle catalysts is also important because the use of multiple metals or alloy nanoparticle catalysts can improve the efficiency of

catalysis and produce more desirable products.

2 Experimental Methods

2.1 Synthesis of Nanoparticles

2.1.1 Materials and sonolysis unit

The Fe nanoparticles were prepared with sonolysis from pentacarbonyliron ($\text{Fe}(\text{CO})_5$, 98% pure) while the Mo nanoparticles were made from hexacarbonylmolybdenum ($\text{Mo}(\text{CO})_6$, 99.5 pure) with the same way. While making the FeS_2 and MoS_2 nanoparticles, sulfur powders were added into the solvent mixed with $\text{Fe}(\text{CO})_5$ and $\text{Mo}(\text{CO})_6$ respectively before sonolysis started. Both Fe and Mo carbonlys were obtained from Sigma Aldrich Chemical Co.

Nanoparticles are fabricated with different solvents: Hexadecane (99%, anhydrous), Peg-400 (Polyethylene glycol 400, 50%) and C_{30} oil (ethyl flo 164, 99+%) . These materials were purchased from Sigma-Aldrich Chemical Co. Argon needed during the process of sonolysis was obtained from Scott Specialty Gases.

The sonolysis synthesis was done by the ultrasonic liquid processor Sonicator 3000 which was from MISONIX, Inc (showed as Fig. 1). The sonolysis unit was set with variable power output of up to 45 W under fixed frequency of 20 kHz. The unit was fitted with a 5-in long half wave extender tip with a probe tip of diameter 0.5” [9]. In order to avoid the possible heat build-up, the power output, processing time and pulsar cycle for cyclic intermittent operation were controlled precisely by the unit.



Figure 1 Ultrasonic liquid processor Sonicator 3000

2.1.2 Synthesis of Nanoparticles

Synthesis of nanoparticles was achieved by sonolysis. As Fig. 2 shows, before the sonolysis started, all of the reactors were put in the reaction vessel which was purchased from Ace Glass, Inc. The vessel was a borosilicate glass 4-neck flask. Its bottom was narrower compared with top, which could make it has the maximum solution in the middle of the flask and let the sonication probe properly immersed in the solution. And it's also necessary to seal the flask by a series of O-rings and greased glass joints to prevent the leakage of gas from the flask and stop the air entering the flask during the sonication.



Figure 2 Experiment set up for sonolysis

- (1) Reaction vessel (2) Water bath (3) Sonication probe**
(4) Temperature controller (5) CO flow tube

Before the start of sonolysis, the solvents in the flask needed to be degassed with argon for 10 minutes, and then the metal carbonyl solution was added in. For synthesis of Iron nanoparticles, the degassed yellow homogeneous solution of $\text{Fe}(\text{CO})_5$ in 70ml solvent was under sonication with the on/off cycle of 4s/1s. As the sonication started, the color of solution turned from yellow liquid to black slurry which showed the progress of the reaction. The temperature of the sonication was controlled around 80C by immersed the flask in a constant temperature bath. The sonolysis was stopped when the conversion of decomposition reached at 85%. After the sonication, hexane was added to the slurry to layer the slurry, and then the black slurry was centrifuged and the upper liquid was decanted, after repeated it 3 times the

black solid remained would get dried. Then the particles were stored for next tests.

For synthesis of Molybdenum nanoparticles, the same process was followed. Mo(CO)_6 was a solid powder. The Mo(CO)_6 powder (2g) was added in 70 ml solvent and yielded a white slurry. During the sonication, it was observed that there were white particles attached along the wall of the vessel which was due to the sublimed Mo(CO)_6 . As the sonication was ongoing, the color of solution turned from clean liquid with white particles to the black slurry. After the sonication, the particles were collected with the same way as in the case of iron.

The metal sulfide nanoparticles were also synthesized with the same procedure. The mole fraction of sulfur and metals in a 2/1 ratio were added before sonication to produce iron sulfide nanoparticle, nano FeS_2 .

All metal and metal sulfide nanoparticle synthesis runs were conducted in three different solvents: hexadecane, Peg-400 and C-30 oil.

2.2 Analysis of nanoparticles

2.2.1 Decomposition Kinetics

During the process of sonolysis, the released gas (CO) was collected and recorded. After the reaction, a plot was drawn to determine the kinetic trends.

2.2.2 Surface Morphology

The prepared black solid nanoparticles were subjected to surface morphology test with SEM (scan electron microscopy) and TEM (transmission electron microscopy). Before the test with SEM and TEM, the solid particles were dried thoroughly. The

TEM images were recorded by FEI BioTwinG² TEM with accelerating voltage of 120 kV, and the SEM images were recorded on the ISI-SX30 SEM with the electron high tension as 20 kV.

2.2.3 XRD

The nanoparticles fabricated from synthesis were dried first. Because of the special requirement of the XRD equipment, the samples needed to be much drier than the morphology test, thus the particles were dried under higher temperature and longer time. Then they were subjected to spectroscopic analysis for characterization. The XRD measurements were taken by a Phillips Vertical Goniometer and CuK radiation ($\lambda = 1.54\text{\AA}$).

3 Results and Discussion

3.1 Decomposition kinetics

The decomposition data were collected from measurement of the CO evolution during sonolysis. The first-order plot of different decompositions can be gained by fitting the CO evolution versus time data, and then the decomposition rate constant of synthesis were calculated. Figures 3, 4 and 5 show the kinetic plots of the $\text{Fe}(\text{CO})_5$ decomposition in three different solvents: hexadecane, Peg-400 and C_{30} oil.. Figures 6 and 7 show kinetic plots of the $\text{Fe}(\text{CO})_6$ decomposition in the presence of sulfur in two different solvents: C_{30} oil and Peg-400.

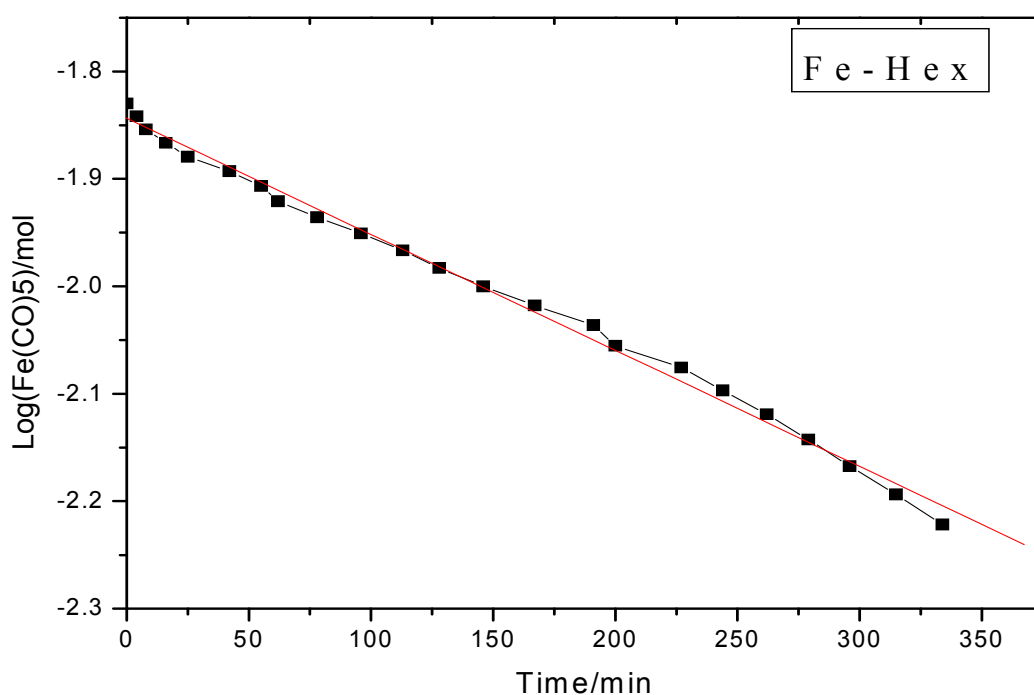


Figure 3. First-order plot for the $\text{Fe}(\text{CO})_5$ decomposition in hexadecane.

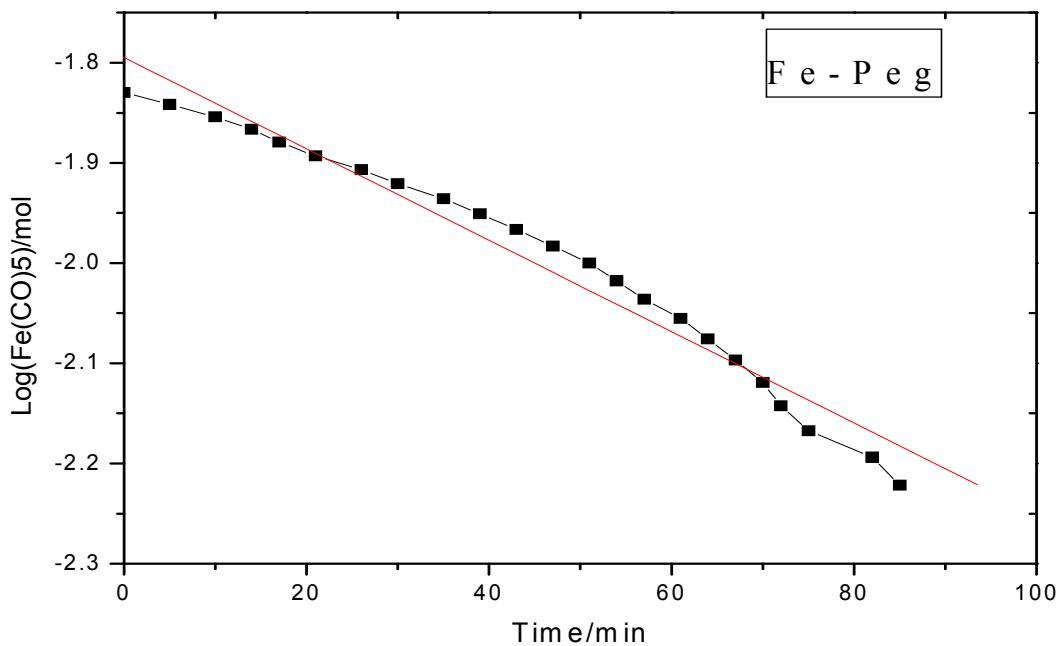


Figure 4 First order plot for the $\text{Fe}(\text{CO})_5$ decomposition in Peg-400.

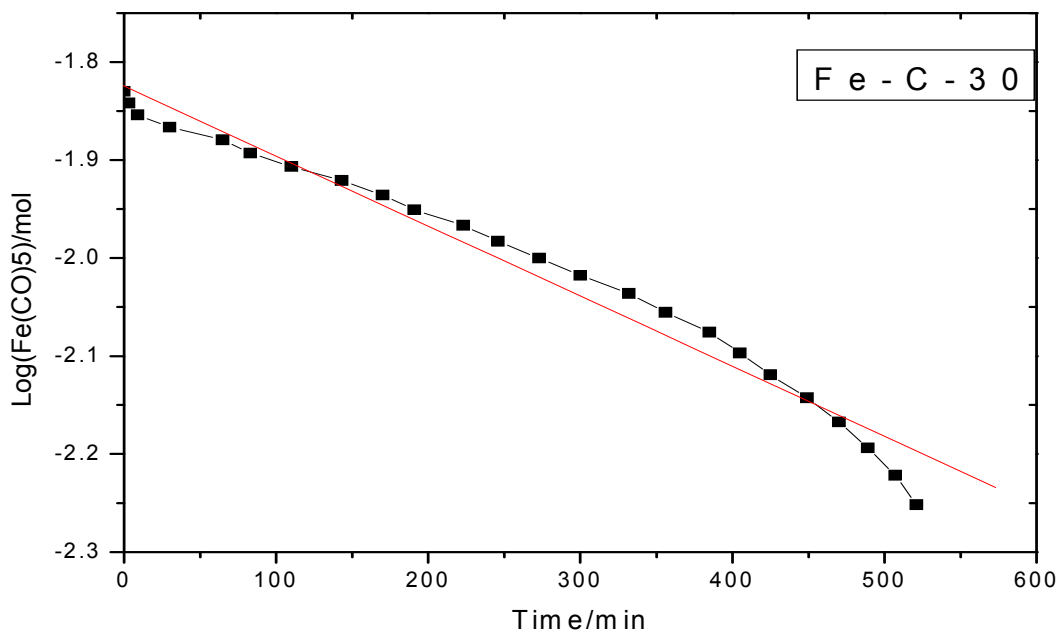


Figure 5. First-order plot for the $\text{Fe}(\text{CO})_5$ decomposition in C-30 oil.

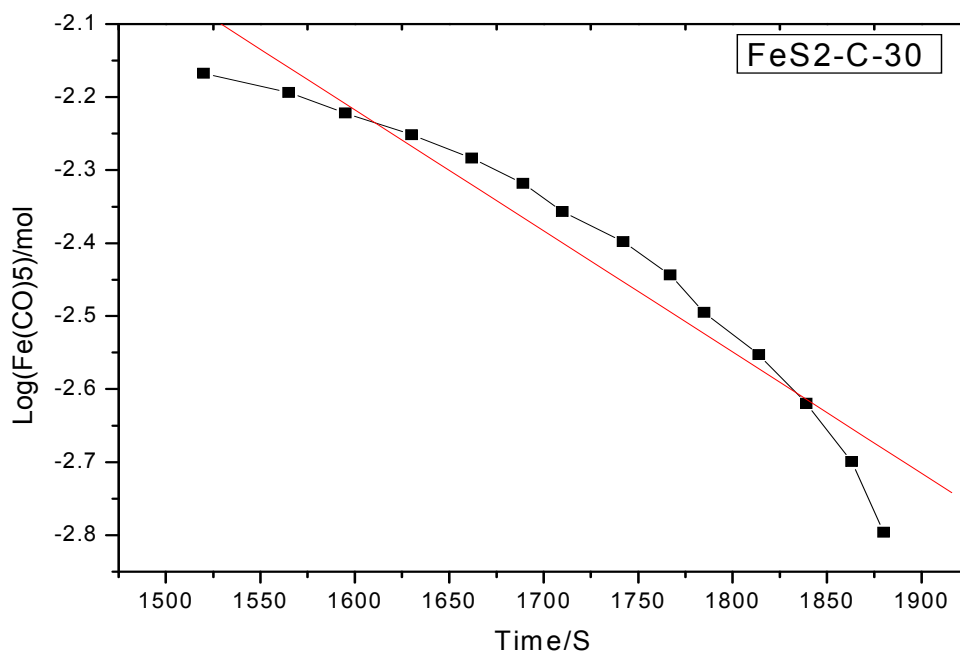


Figure 6. First-order plot for the Fe(CO)₅ decomposition with sulfur in C-30.

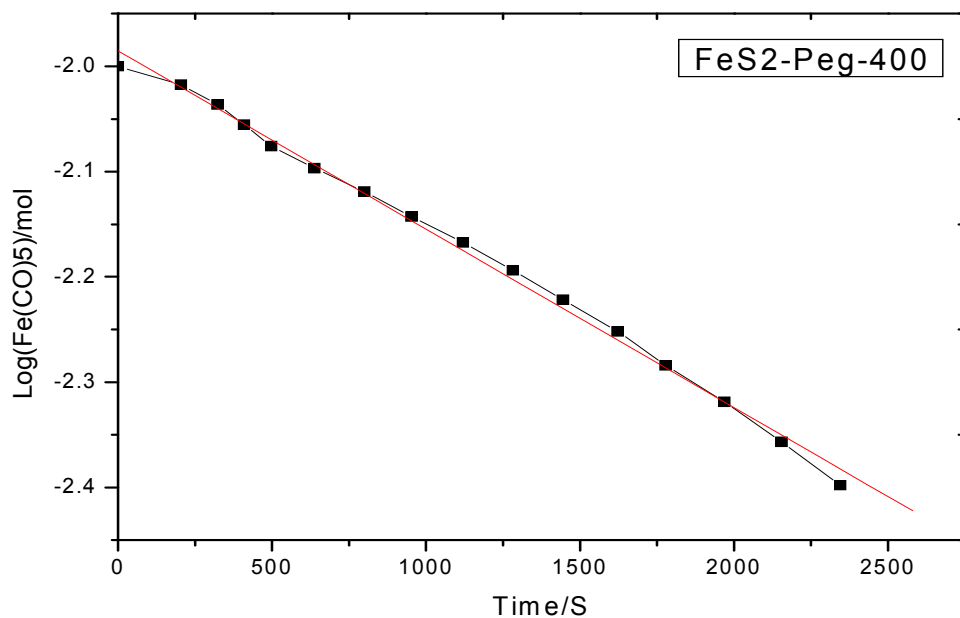
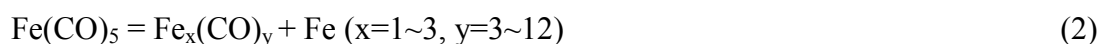


Figure 7. First-order plot for the Fe(CO)₅ decomposition with sulfur in Peg-400.

Similar plots were obtained for Mo.

The complete decomposition of $\text{Fe}(\text{CO})_5$ produces 5 moles of CO per mole of Fe.



Thus, from the gas collected data, the extent of decomposition can be calculated.

From Figures 3 - 5, the decomposition reaction data were extracted and shown in Table 1. There has been study about the effect of different hydrocarbon solvents on decomposition rate of $\text{Fe}(\text{CO})_5$, which showed that the rate constants were 8.0, 5.5, 5.1 and $0.7 \times 10^{-2} \text{ min}^{-1}$ at 25°C in decalin, decane, nonane and octane solvents respectively [19]. The k values showed in Table 1 were about two orders of magnitude lower than those reported above.

The difference in rates between the present and those previously reported in literature can be attributed to different solvents used in the two studies. All calculated values from the present study are listed in Table 1.

Table 1 Effect of reaction parameters during $\text{Fe}(\text{CO})_5$ decomposition

Precursor		Additive	Solvent	Time/min	Conversion	$ k /\text{min}^{-1}$
$\text{Fe}(\text{CO})_5$	2ml (0.015mol)	None	Hexadecane	480	85%	10.8E(-4)
		None	Peg-400	110	85%	45.6E(-4)
		None	C ₃₀ Oil	598	85%	7.15E(-4)
	0.01mol (0.02molS)	S	Hexadecane	40	85%	-----
		S	Peg-400	53	85%	1.69E(-4)/S ⁻¹
		S	C ₃₀ Oil	31	85%	17.0E(-4)/S ⁻¹

It can be concluded that when the 3 different solvents were compared, the reaction rate of synthesis of Fe nanoparticles followed: Peg-400 > Hexadecane > C₃₀ Oil. The

reaction rate of synthesis of FeS₂ nanoparticles followed: C₃₀ Oil > Hexadecane > Peg-400. To explain the difference of rates in solvents, it's necessary to know the different properties of these solvents. Hexadecane consists of 16 carbon atoms with a formula of C₁₆H₃₄. Peg-400 is a glycol with it's a formula C_{2n}H_{4n+2}O_{n+1} and it's very hydrophilic. C₃₀ oil (also called ethyl flo- 164), is a 1-decene homopolymer liquid with a formula of [CH₂CH[(CH₂)₇CH₃]]_n. The difference in rates of synthesis of nanoparticles is a complex issue.

Figures 8 – 10 depict plots of for the Mo(CO)₆ decomposition in three different solvents. Figure 11 shows a plot for Mo(CO)₆ in the presence of S.

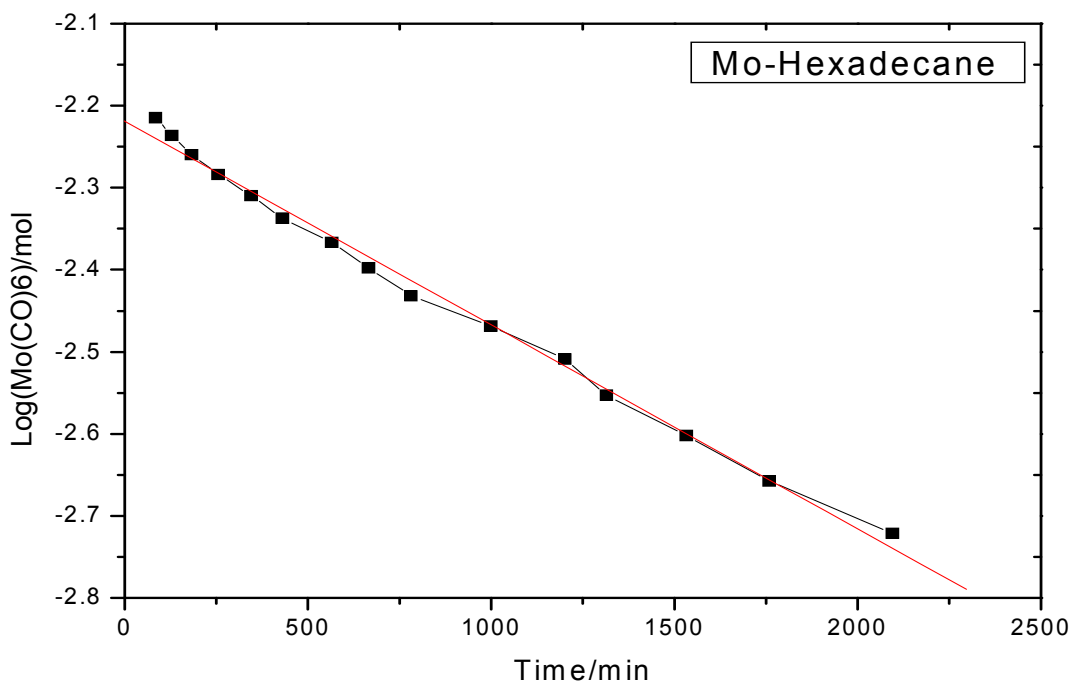


Figure 8 First-order plot for the Mo(CO)₆ decomposition in hexadecane.

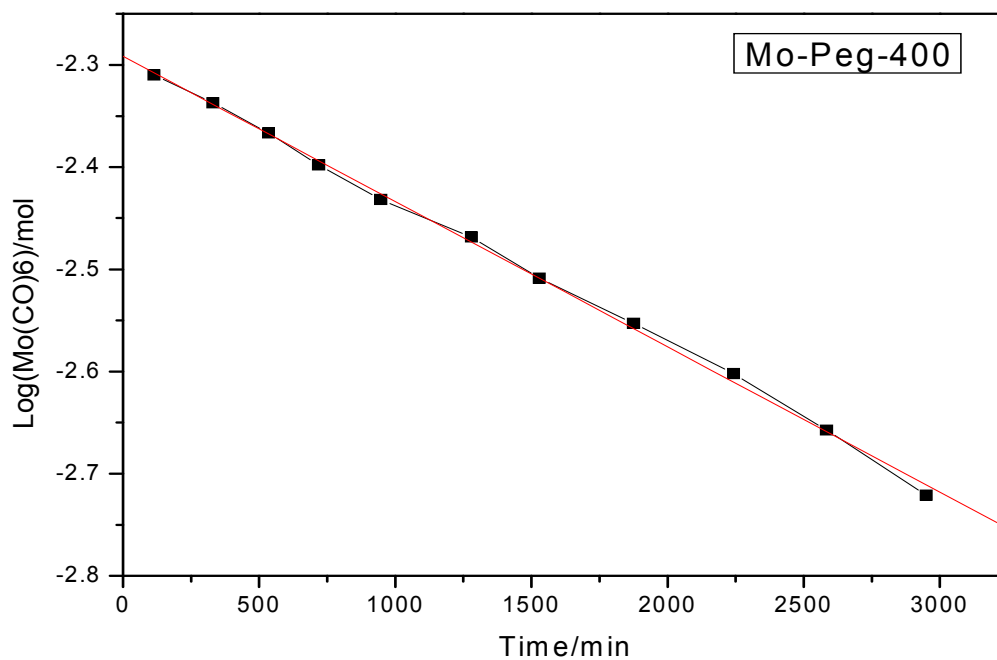


Figure 9 First-order plot for the Mo(CO)₆ decomposition in Peg-400.

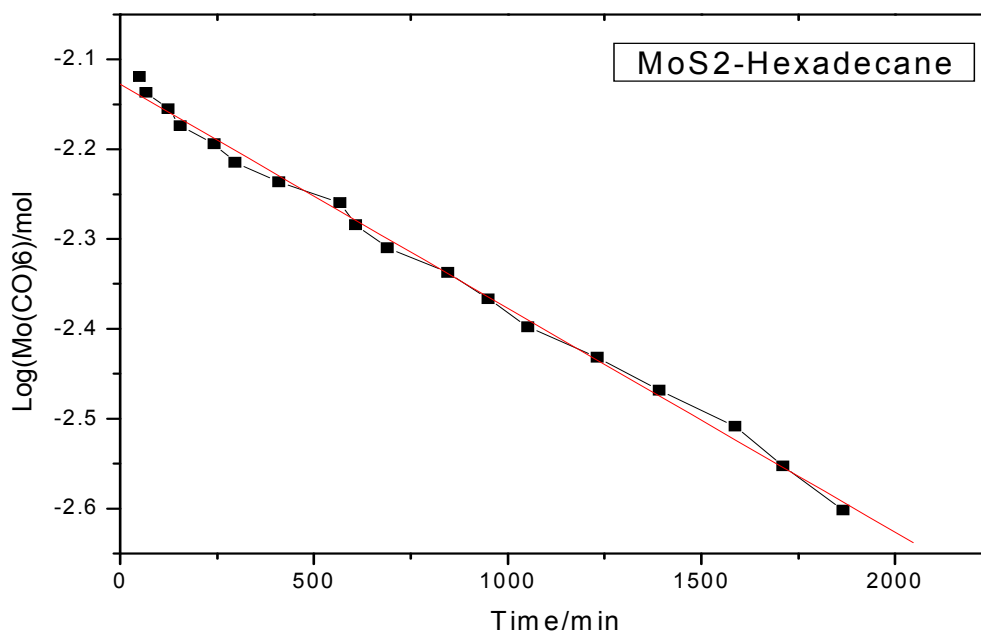


Figure 10 First-order plot for the Mo(CO)₆ decomposition with sulfur in hexadecane.

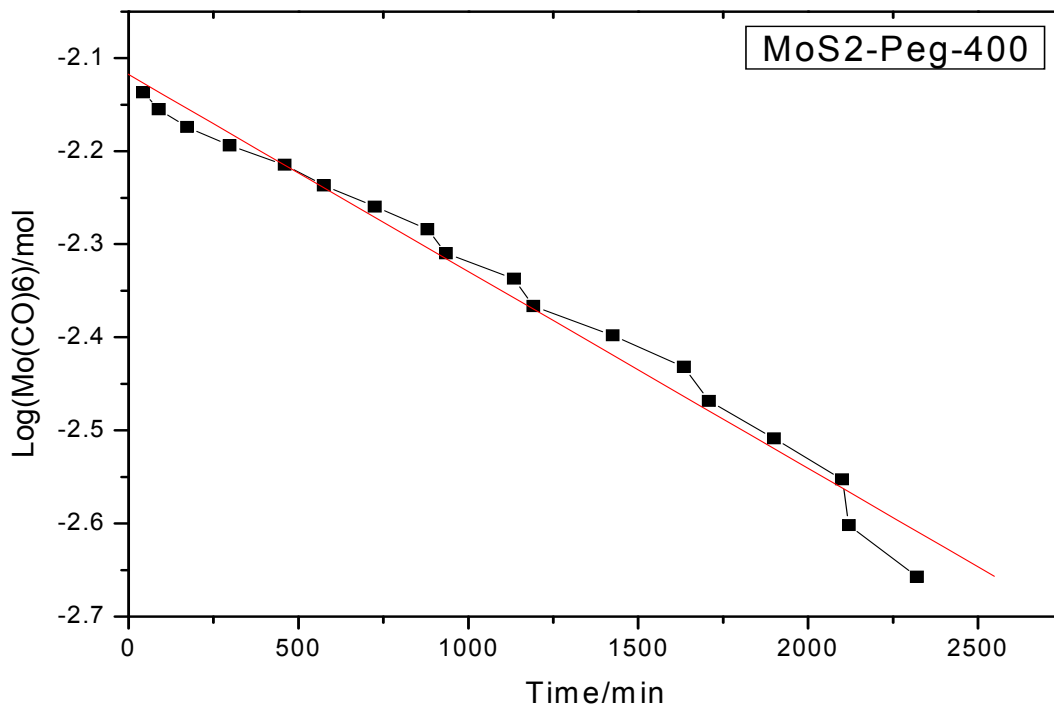


Figure 11 Fig First-order plot for the Mo(CO)₆ decomposition with sulfur in Peg-400.

From Figures 8 - 10, the data for decomposition of Mo(CO)₆ are shown in Table 2. It can be concluded that for the 3 different solvents, the reaction rate follows: Hexadecane > Peg-400. For the reaction rate of synthesis of MoS₂ nanoparticles, the order was: Hexadecane > Peg-400.

Table 2 Effect of reaction parameters during Mo(CO)₆ decomposition

Precursor		Additive	Solvent	Time/min	Conversion	k /min ⁻¹
Mo(CO) ₆	2g (0.008mol)	None	Hexadecane	2095	85%	2.48E(-4)
		None	Peg-400	2950	85%-	1.42E(-4)
		None	C ₃₀ Oil	-----	-----	-----
	0.01mol (0.02molS)	S	Hexadecane	1865	85%	2.5E(-4)
		S	Peg-400	2320	85%	2.12E(-4)
		S	C ₃₀ Oil	-----	-----	-----

When the decomposition of iron and molybdenum nanoparticles are compared, we can see that Mo(CO)₆ decomposed slower than Fe(CO)₅: this could be explained as follows: first, Mo(CO)₆ is solid powders and only slightly soluble while Fe(CO)₅ is liquid and is miscible with solvents like hexadecane. Second, the Mo-CO bond has the strength as 150.3 kJ/mol while the Fe-CO bond has 116.0kJ/mol which shows the Mo-Co bond is stronger and harder to break [9,20]. Also it is clear that synthesis of metal sulfide nanoparticles form faster than metal nanoparticles. This is due to the presence of sulfur that reacts with Mo nanoparticles as soon as these are produced. . In the rich sulfur environment, it is thermodynamically favorable for the metals to be converted to metal sulfide [21].

3.2 SEM and TEM of nanoparticles

The nanoparticles fabricated from the synthesis were dried and their surface morphology was observed by SEM and TEM.

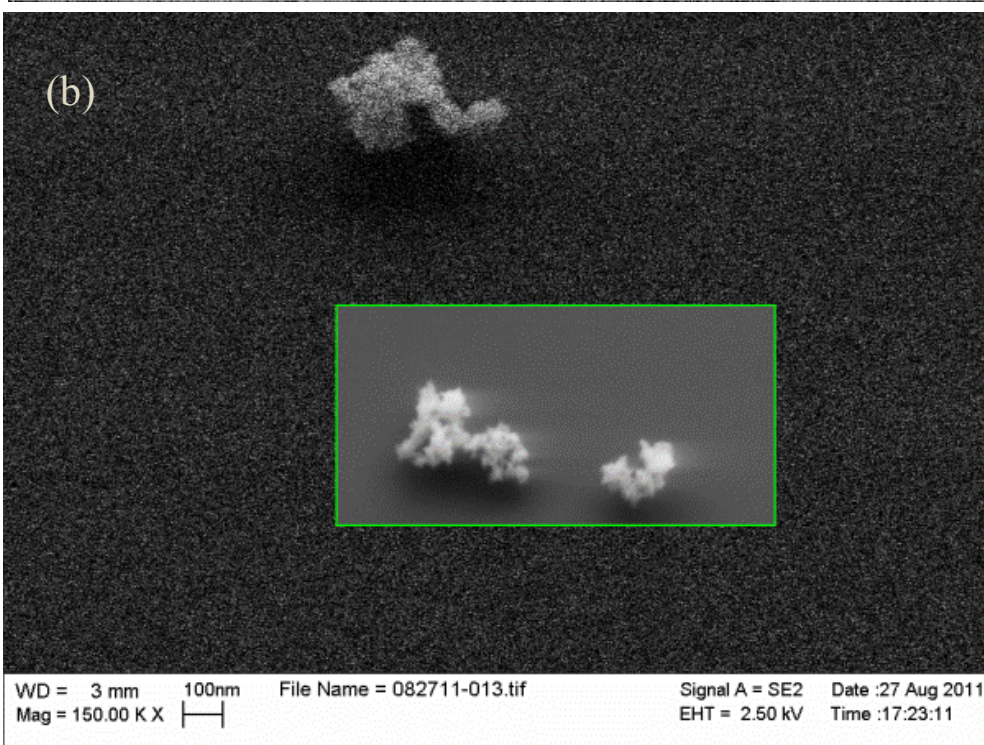
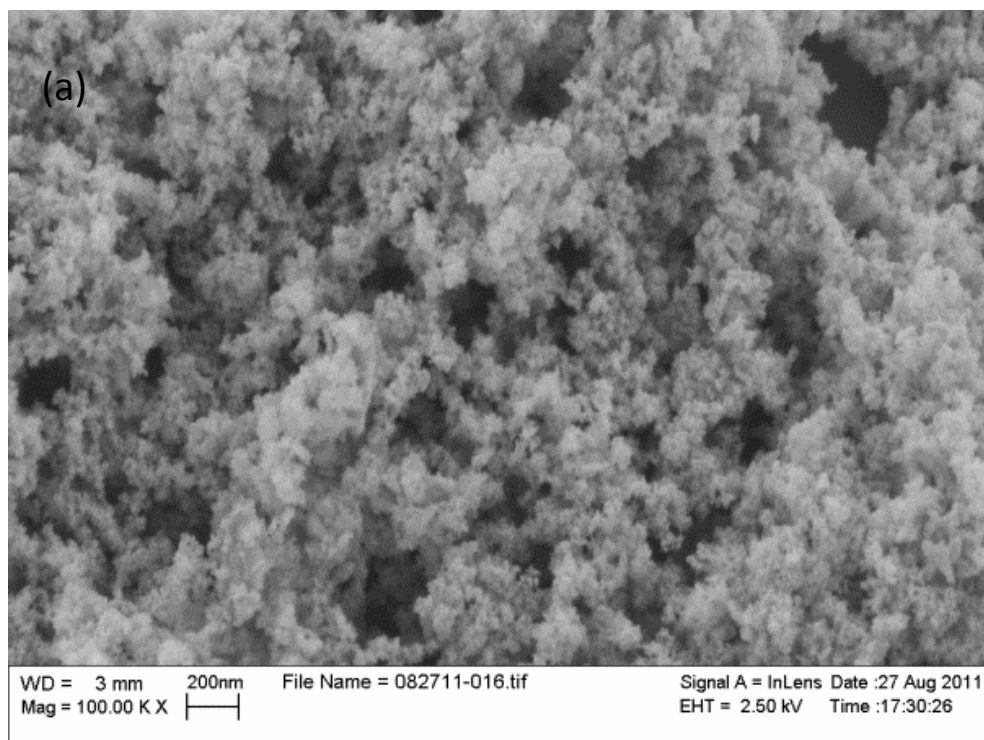


Figure 12 SEM of Fe Nanoparticles made in Hexadecane
(a) 100 K (b) 150K

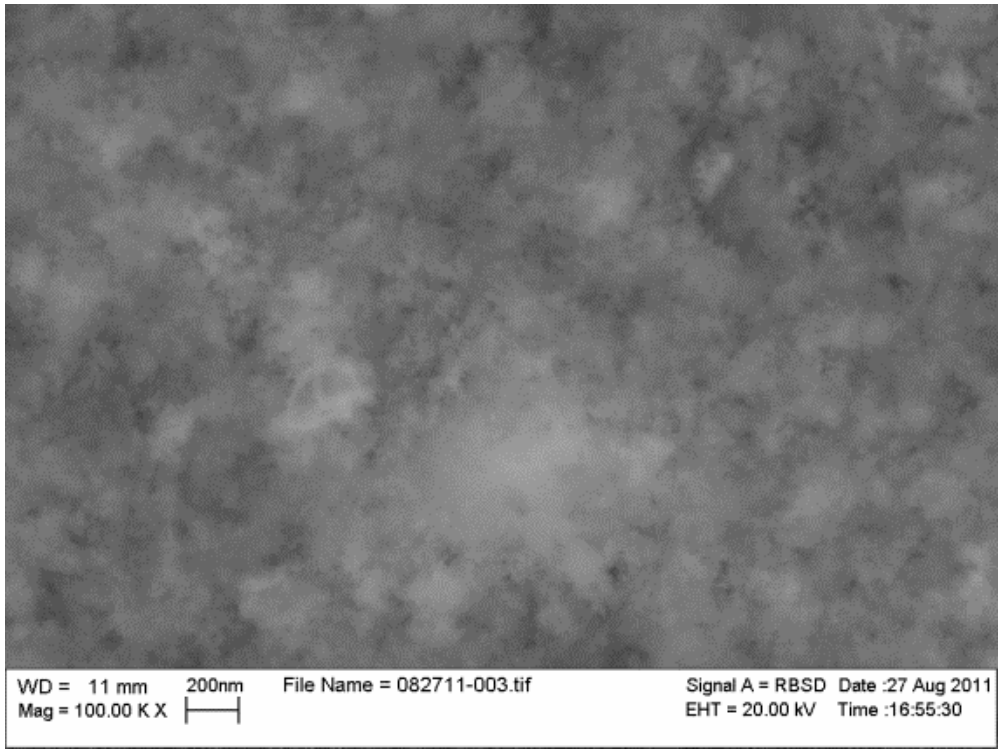


Figure 13 SEM of Fe Nanoparticles made in Peg-400

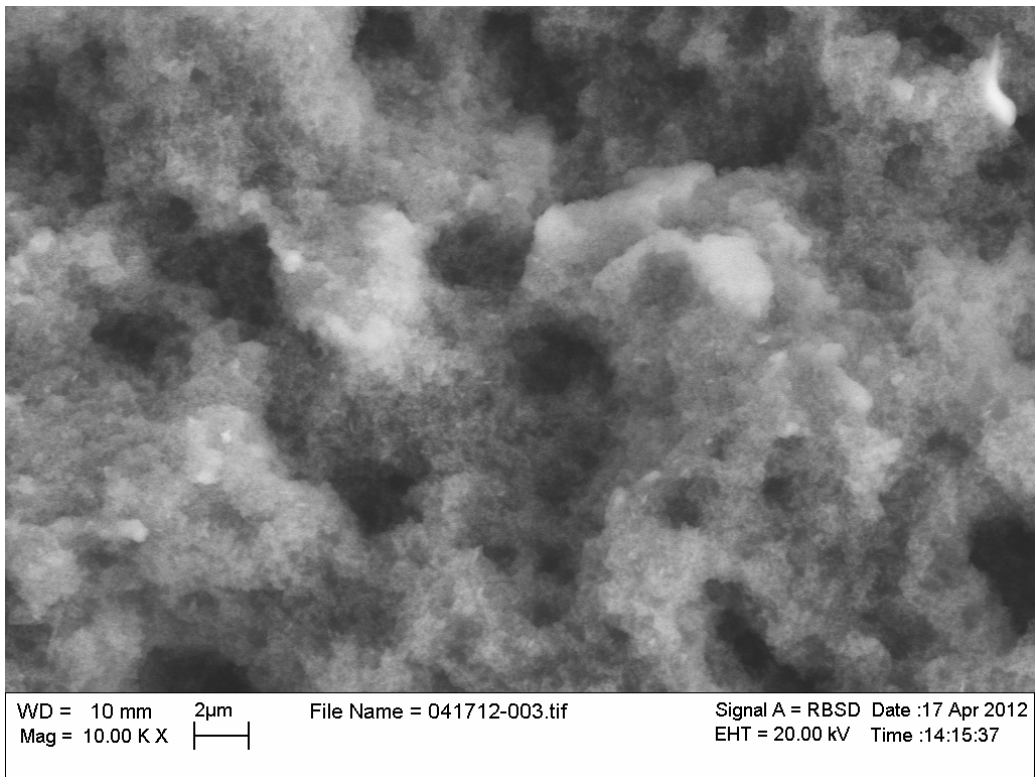


Figure 14 SEM of Fe nanoparticles made in C₃₀ oil

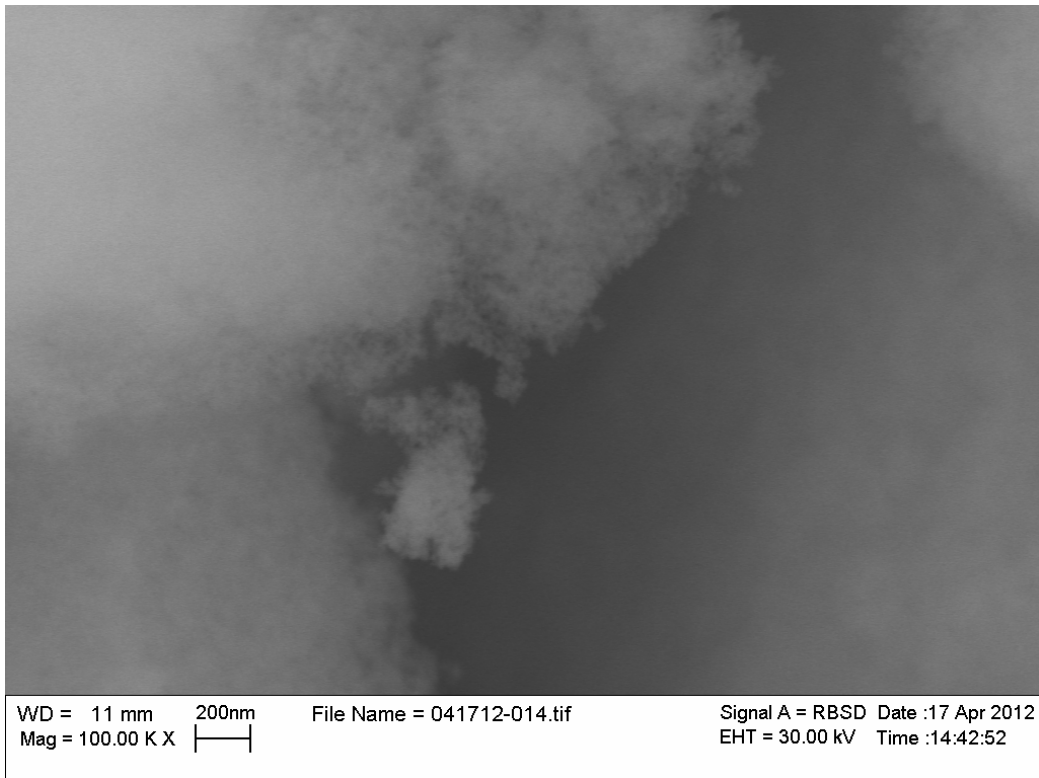


Figure 15 SEM of Mo nanoparticles made in Hexadecane

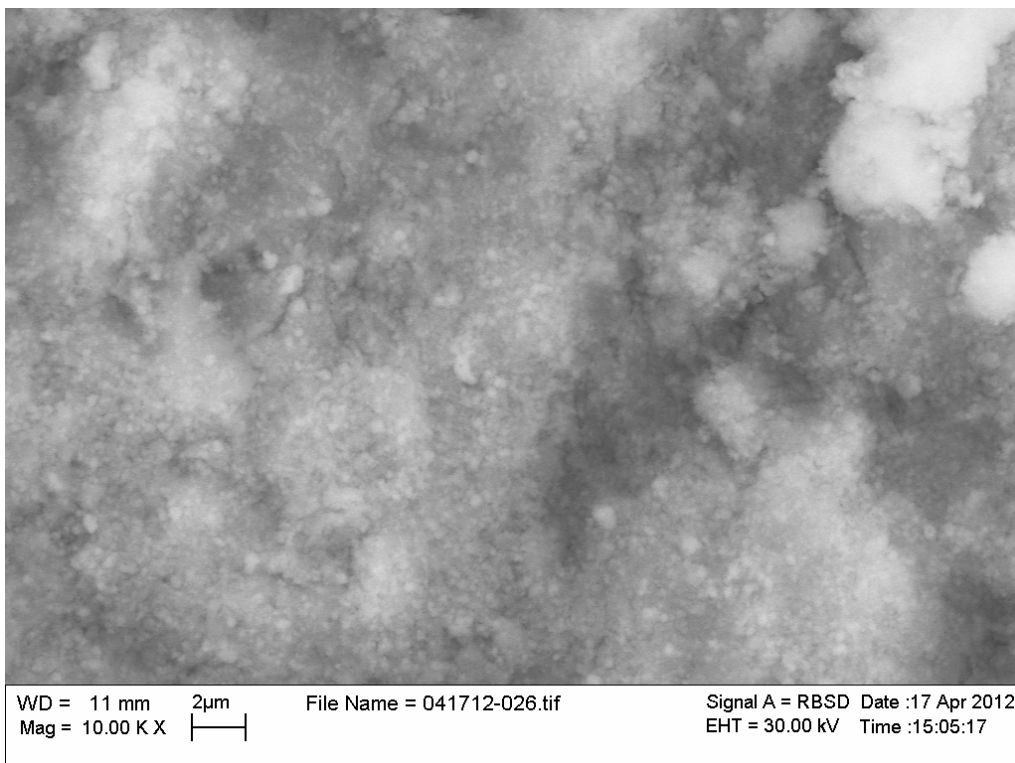
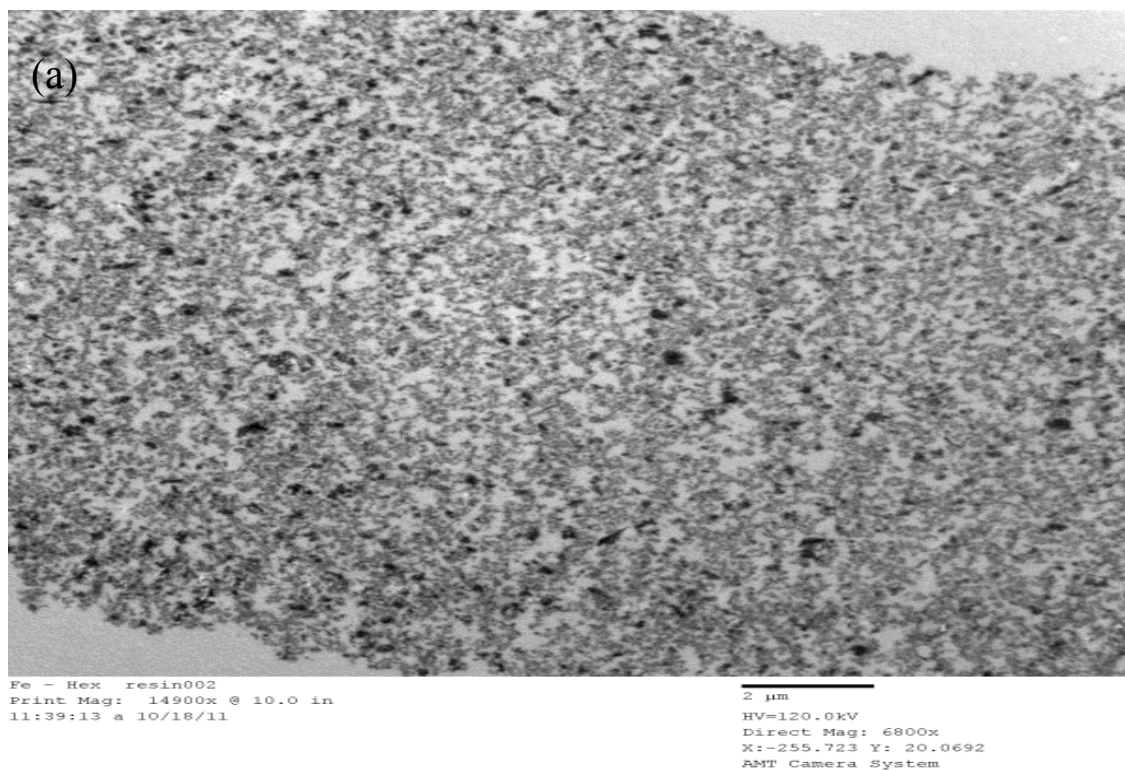


Figure 16 SEM of MoS₂ nanoparticles made in hexadecane

From the SEM pictures of different nanoparticles, we noticed that the nanoparticles were congregated to form large aggregates. This could be attributed to the high activity of nanoparticles. Also because the nanoparticles were rinsed and dried before the test, it could be also possible that the rinse solution or the solvents left were still attached on the surface of nanoparticles, which could glue them together.

We also see that the nanoparticles made in hexadecane has the best morphology, they are not as aggregated as particles made in other solvents. This also supports the presumption that the solvents left may attach on the surface of particles and congregate them together to aggregates, because Peg-400 and C₃₀ oil are liquids that are stickier compared with hexadecane. The SEM results also show that the Mo nanoparticles are more aggregated than Fe nanoparticles which may imply the Mo nanoparticles have higher activity than iron.



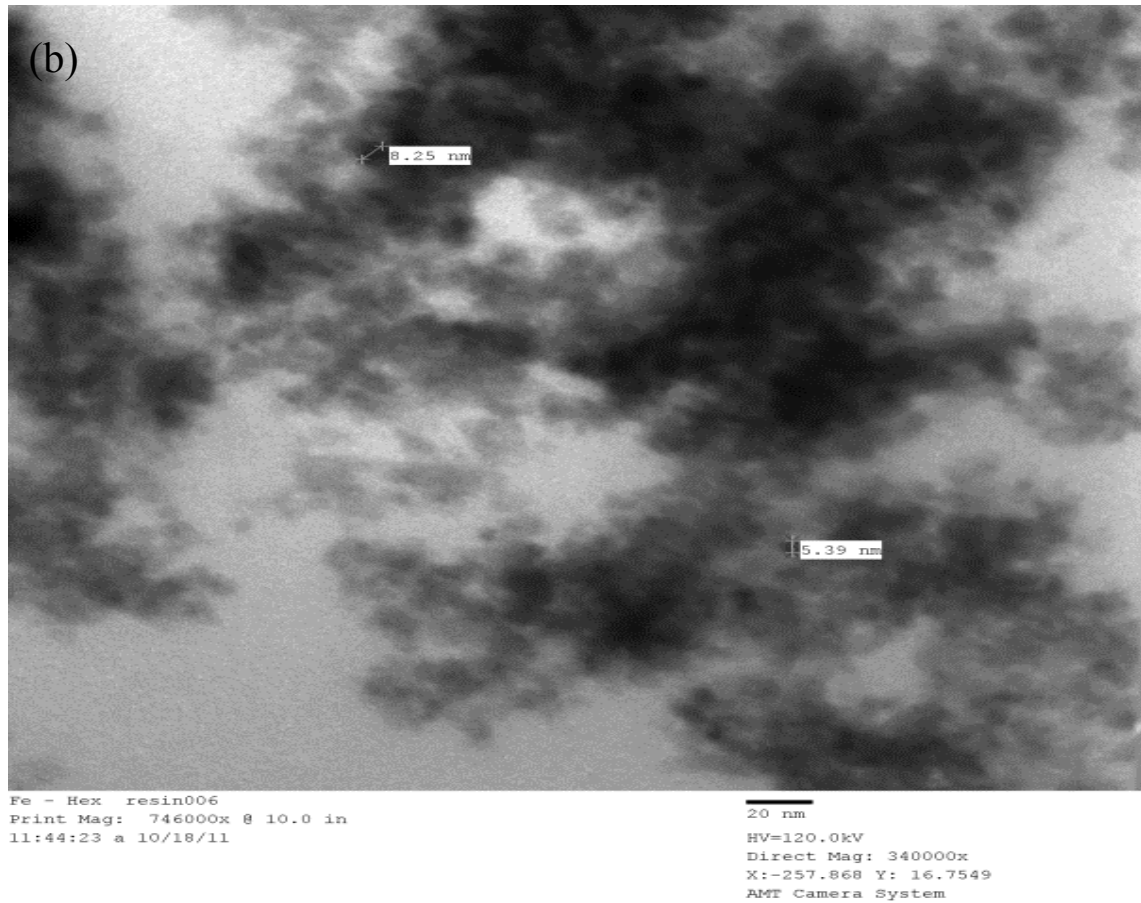


Figure 17 TEM of Fe nanoparticles made in hexadecane

(a) Without particle size (b) With particle size

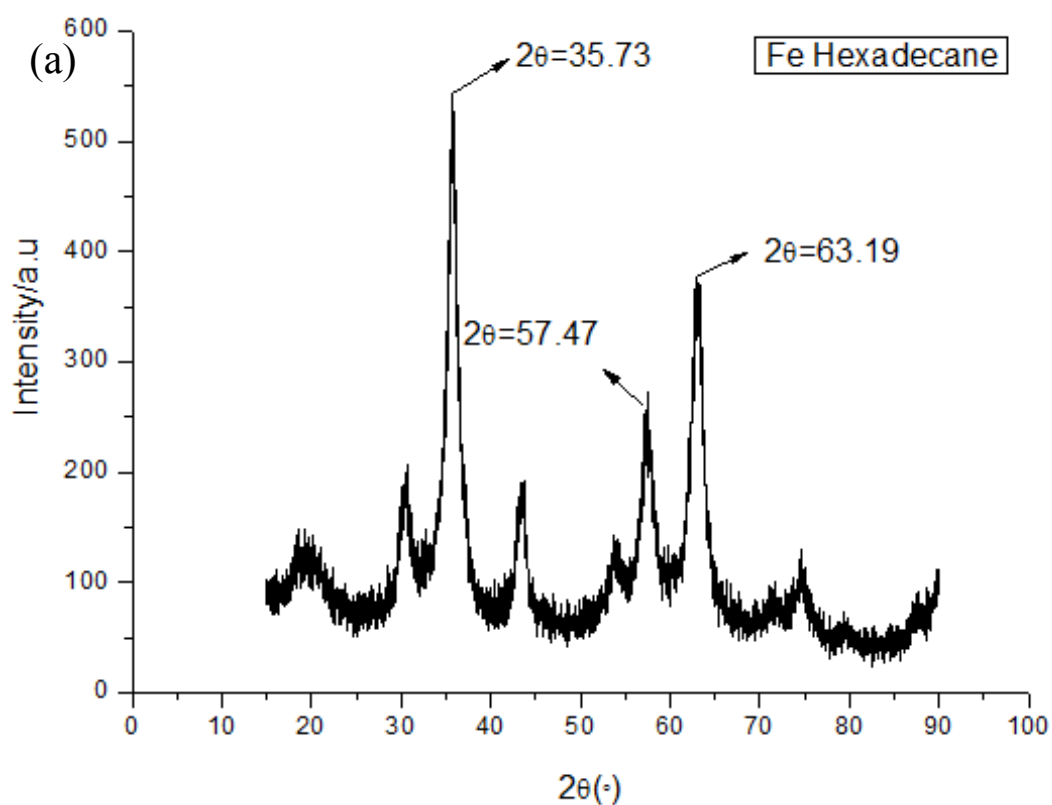


Figure 18 TEM of FeS₂ nanoparticles made in hexadecane

(a) Without particle size (b) With particle size

From the TEM images, it is seen that the particle size of Fe and FeS₂ nanoparticles is on the scale of 10nm. Same was observed with the SEM images: most nanoparticles were stuck together likely due to either the inherent tendency of nanoparticles to aggregate or the sample was not thoroughly rinsed before the measurements..

3.3 XRD analysis of nanoparticles



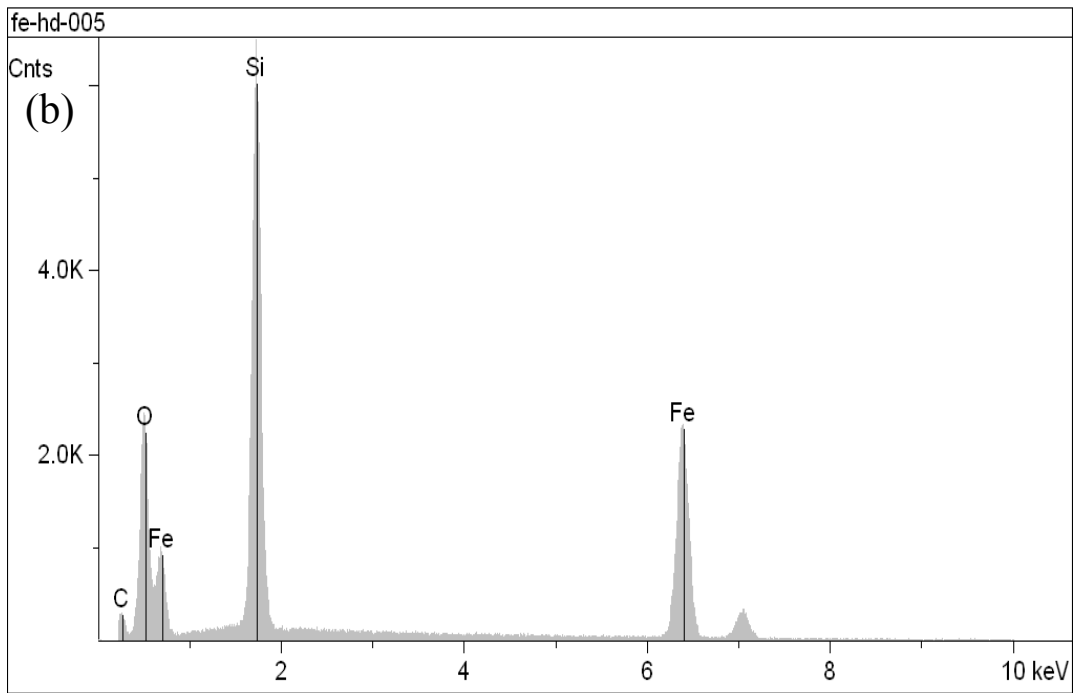
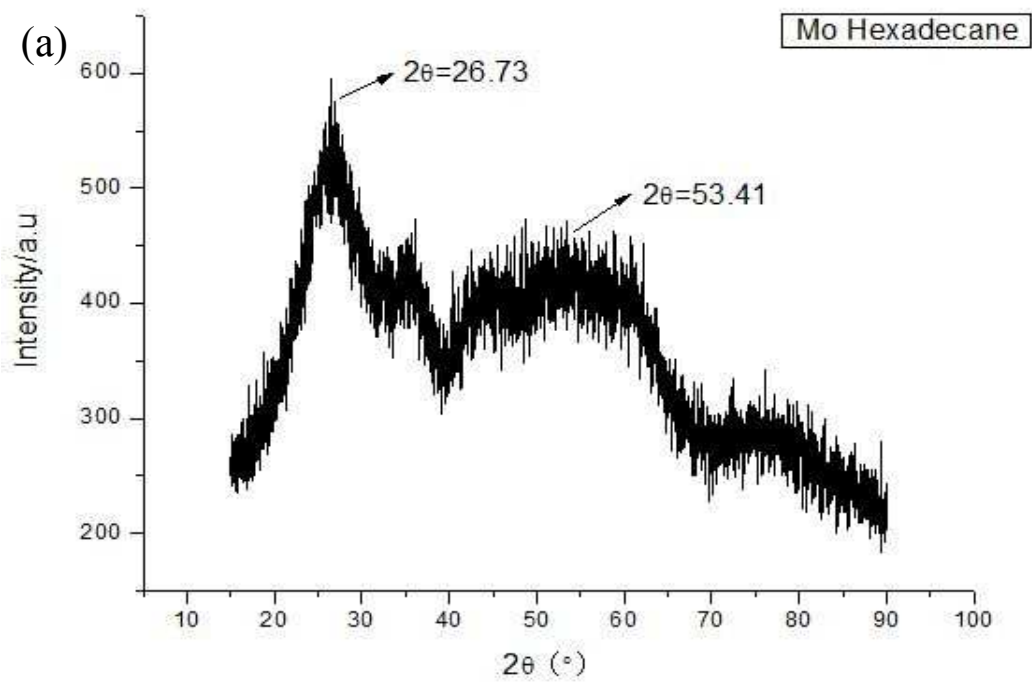


Figure 19 XRD (a) and EDS (b) of Fe nanoparticles made in Hexadecane



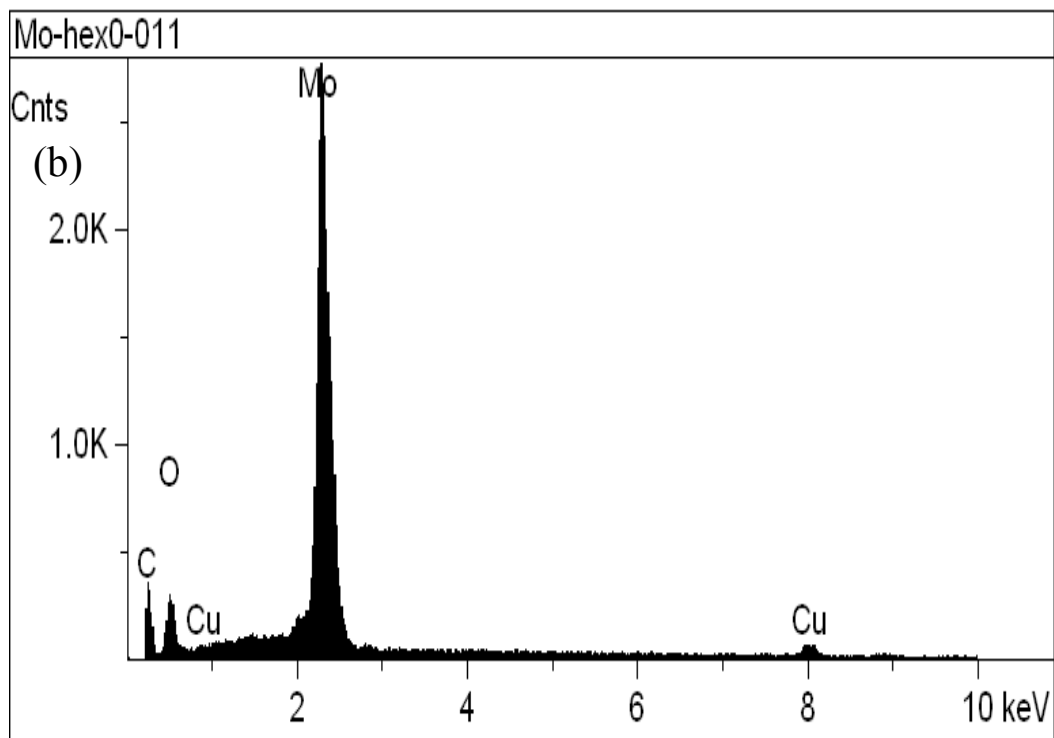


Figure 20 XRD (a) and EDS (b) of Mo nanoparticles made in Hexadecane

The results of XRD spectrum of iron and molybdenum nanoparticles are shown in Figures 16 and 17. The spectrum was analyzed by JADE software. From the database, we found that the peaks of iron samples at 2θ indicate the presence of Fe_2O_3 , likely FeO and iron carbide, as verified by EDS. Combined with the XRD spectrum and EDS of molybdenum particles, it can be assumed that the Mo nanoparticles may be molybdenum oxide and molybdenum carbide. During the analysis of Mo nanoparticles, there is no exact match of the peak of spectrum with database, this may be because the particles are too small and they are amorphous.

4 Conclusion:

Nanoparticles of iron and molybdenum both metallic and sulfide, were synthesized in different solvents. After the analysis of decomposition kinetics, and characterization data, the following conclusions were drawn.

1. Synthesis of iron nanoparticles was faster than molybdenum, and the additive of sulfur accelerated the sonolysis reaction. In all cases, 85% decomposition of the starting metal carbonyl was achieved. The impact of different solvents on the decomposition kinetic varied among different nanoparticles. For iron nanoparticles, the solvent dependence was in the order: Peg-400 > C₃₀ oil > Hexadecane. For sulfide iron synthesis, the rank was reversed. For synthesis of molybdenum and molybdenum sulfide nanoparticles, sonolysis exhibited the same behavior in two solvents: Hexadecane > Peg-400.

2. The surface morphology showed that the particles were congregated, which may be due to the nature of nanoparticles and insufficient rinsing before the test. It was also observed that the particles made in hexadecane were smaller than those in other two solvents.

3. The XRD analysis showed that the main components of the iron system were nanoparticles were Fe₂O₃ and other iron oxide phases, though some iron carbide particles was also detected. The components from the molybdenum reaction were molybdenum oxide and carbide particles.

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