Microwave assisted synthesis of cadmium carbide

Dissertation submitted to

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For the partial fulfillment of the award of M.Sc. (Honours) Chemistry

by

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Declaration

I hereby declare that the Project report entitled, “Microwave synthesis of cadmium carbide” submitted for M.Sc. (Honours) chemistry degree is entirely my original work and all the ideas and references have been duly acknowledged. It does not contain any work for the award of any other degree at any university.

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Acknowledgement

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I would also extend my sincere thanks to Lovely Professional University for all the facilities required for this project. This includes, facilities for laboratory, library, accommodation and neat and clean and green campus. Above all, the great thanks to the author of the different papers that I have taken into consideration during my project work.

Content

Chapter 1

Introduction

1.1 Background of the project: In the synthesis of materials microwave (MW) is gaining a great importance. Microwave assisted synthesis is much faster, cleaner and economical than the conventional heating (CH) method. A number of materials such as carbide, silicide, zeolites and complex oxides have been synthesized with the help of domestic microwave oven (DMO). Most of these are of technological and industrial importance. Here we are going to synthesize and characterized cadmium carbide.

1.2 Microwave Heating and conventional Heating: Microwave assisted synthesis offers a number of advantages over conventional heating (CH) method such as (1) non-contact heating (2) energy transfer instead of heat transfer; (3) rapid heating (4)selective material heating; (5) volumetric heating; (6) quick start-up and stopping (7) heating from interior of material body; (8) higher level of safety and automation. Due to these advantages microwave (MW) are used in order to heat different substances. MW are also used for food processing, sterilization and pasteurization. Microwave (MW) chemistry is based upon the efficient heating of matter by microwave dielectric heating (MWDH), i.e., on the ability of a specific material to absorb microwave (MW) energy and to convert it into heat. Microwaves (MW) are the electronic waves having frequencies ranging from 0.3 GHz to 300 GHz and with wavelengths of between 1 mm and 1 m, which are between infrared and radio frequency waves in the electromagnetic spectrum as shown in figure 1.1 [1,3]

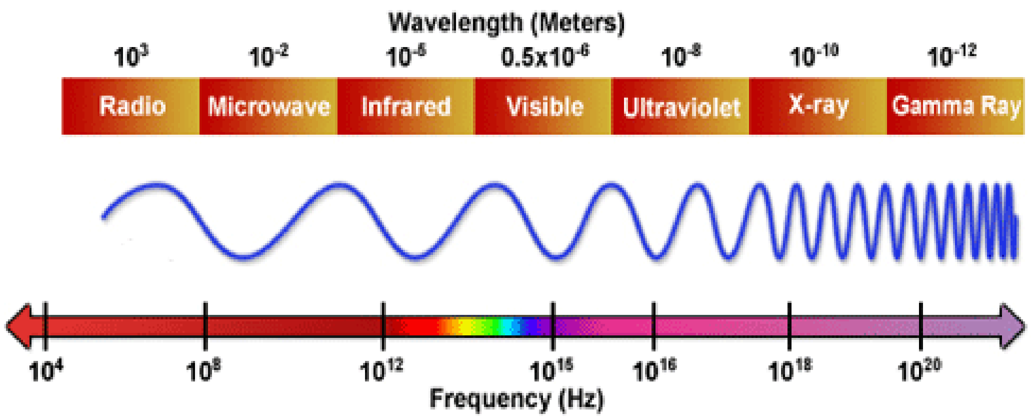

Figure 1.1 Electromagnetic spectrum

The commonly used frequency in laboratories and homes for MW heating is 2.45 GHz (with a wavelength of about 12.24 cm).

In contrast, the conventional heating (CH) usually involves the use of an electric furnace or oil bath, which heats the walls of the reactor and then the reactants by convection or conduction. Microwave (MW) heating is the transfer of electromagnetic (EM) energy to thermal energy (TE) and it is the energy conversion rather than heat transfer. Since microwaves can penetrate throughout the volume of material resulting in volumetric heating. Hence, it is possible to achieve rapid and uniform heating of thick materials.

1.3 Carbides: Carbide is a chemical compound in which carbon is combined with a metallic or semi-metallic element. Carbides are the compounds in which anion in one or more carbon atom. Most of the metals form carbides but not all. In chemistry, carbide is a compound composed of carbon and a less electro negative element. Carbides can be generally classified by the chemical bonds type as follows

1) Salt like or ionic carbides
2) Covalent carbides
3) Interstitial carbides

4) Intermediate transition metal carbides

1.4 Properties of Carbides: (1) Carbides are hard, resistant to heat and corrosion which makes them excellent candidate for coating of drills and other tools.

(2) They have electric conductivity.

(3) They have thermal expansion and abrasiveness.

1.5 Cadmium Carbide: Cadmium carbide is available in various forms and shape such as ingot, foil and plate. In the high pure form it is present as carbide powder nanoscale and submicron powder, single crystal and polycrystalline form. Since it is very difficult to make cadmium carbide and a very high temperature can be achieved through microwave heating, efforts will be made to synthesize this compound through microwave heating.

Chapter 2
Literature review

2.1 History of microwave heating: The time period from 1935-1945 includes World War II and it was a very difficult time for the people of the Earth [1]. Radar played a very important role in World War II and these years are the golden years for advancement in microwave technology. The major microwave developments occurred at MIT Radiation laboratory in America. The microwave heating effect was accidentally discovered in 1945 by Percy Le Baron Spencer. He was an American engineer and inventor. During his working on Radar applications of microwaves, he found that a chocolate bar melted in his pocket [2, 5]. He realized that the microwaves he was working with had caused it to melt. After experimenting, he realized that microwave would cook food quickly even faster than conventional ovens that cook with heat. The Raytheon Corporation produced the first commercial microwave oven in 1954.

2.2 Mechanism of microwave heating: Microwave heating is based upon the microwave dielectric heating effect of materials. It depends upon the ability of a material or solvent to absorb the microwave energy and convert it into heat. The ability of the material to interact with microwave is dependent on the dielectric constant and dielectric loss. The dielectric constant is a measurement of ability of the sample to obstruct the microwave energy when it passes through it and the dielectric loss measures the ability to dissipate that energy in the form of heat. The effect of microwave heating also depends upon the nature of the solvent. The solvent should be capable of absorbing the microwave energy. The solvent should also be capable of converting the microwave energy into heat so that the efficiency of the conversion process depends upon the dielectric loss factor. If the value of dielectric constant is more, then the material or the solvent will more efficiently heated and absorb the microwave radiations. Molecules tend to align with the microwave interacting with the body. If the dielectric constant is very less or low then the material or the solvent will be unable to interact or couple with the microwave radiations. If the rate of flow of heat inside the heating block is restricted then there is slow heating. To increase this rate of heat flow we can use microwaves. The commercial microwave involves the safety issues regarding the high temperature and pressure, the sample can burn and the container may rupture. So we should use the container having high temperature tolerance.

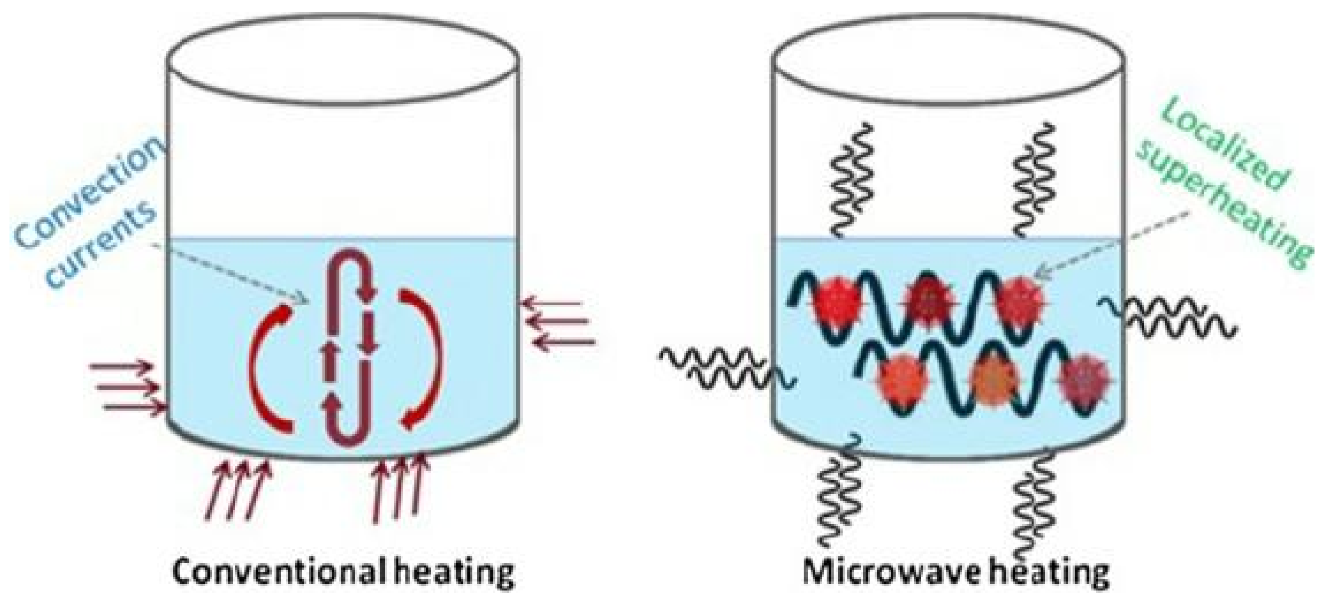

Figure 2.1: Microwave heating verses conventional heating

Conventional heating: A comparison with conventional heating method would provide us a base to compare the differences between the microwave heating and conventional heating and to know about the advantages of microwave heating. In conventional heating, the heat transferred to the volume of the sample is utilized to increase the temperature of the surface of the container and then the internal material. It is known as "wall heating". As a result a large amount of energy supplied through conventional energy source is lost to the surrounding. In conventional heating method the rate of heat transfer depends upon the following factors:

(1) Density of the material

(2) Specific heat of the material

(3) Thermal conductivity

These all three factors give rise to thermal diffusivity.

The microwave heating mechanism is explained as following:

2.2.1 Dipolar polarization: The phenomenon which is responsible for the most of the microwave heating effects observed in case of the solvents is known as dipolar polarization. For instance, in case of water the individual atoms have different electronegativity which results in the permanent electric dipole on the molecule [8]. The dipoles will tend to align themselves with rotation, and the energy for this rotation will be provided by the electric field. The dipole is sensitive to the electric field. This alignment of the molecules is very fast in case of free molecules, but in case of liquids it is prohibited by the other molecules. So the ability of the molecules to align in the electric field is limited which affects the behavior of molecules having different frequencies in the electric field.

Under low frequencies the overall heating effect is very small because the dipole may react in the phase by aligning itself in the electric field. While some of the energy is gained by the molecules and some energy it lost during the collisions, as a result the heating effect is not that much. On the other hand in case of high frequency electric field, the dipoles do not rotate because they do not have the time to respond to the electric field. Since there is no motion in the molecules, so no energy transfer takes place therefore, no heating.

In case of microwave oven the frequency is low and the molecules can rotate and the heat can be produced.

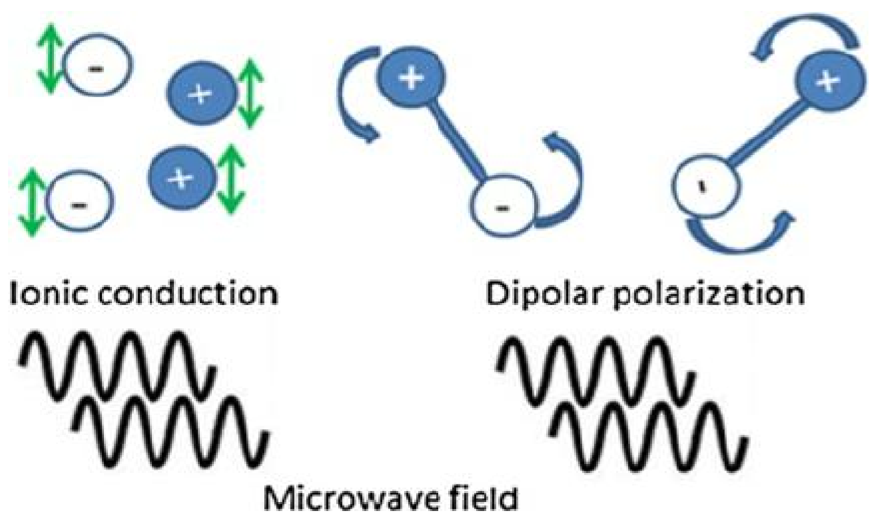

Figure 2.2.1 : Dipolar polarization verses conduction effect

2.2.2 Conduction effects: Conduction has more effect in heating mechanism than the dipolar polarization. Many materials show losses due to conduction in the presence of microwave radiations. Addition of dissolved salts in water affects the dielectric properties as there is increase in conduction [8]. In case of majority of the solids the dielectric losses are arises due to these conductions and can be strongly affected by the temperature. In case of metals and metal powder the heating is dependent on the conduction losses.

2.2.3 Interfacial polarization: Theoretically and experimentally it is known that a heterogeneous structure of material gives rise to a dielectric dispersion which is known as the interfacial polarization.

2.3 Single mode or multimode microwave cavity: Generally the multimode cavity is used as an applicator in microwave processing because of low cost, adaptability to wide range of heating loads and simple construction [3]. A multimode cavity mode also provides a uniform distribution of electric field. Multimode cavity can also be used for large size objects and it is suitable, therefore it is used in most of the industrial microwave processing systems. Single mode applicators are used in specific applications such as joining of ceramics and laboratory scale study of microwave and material interactions.

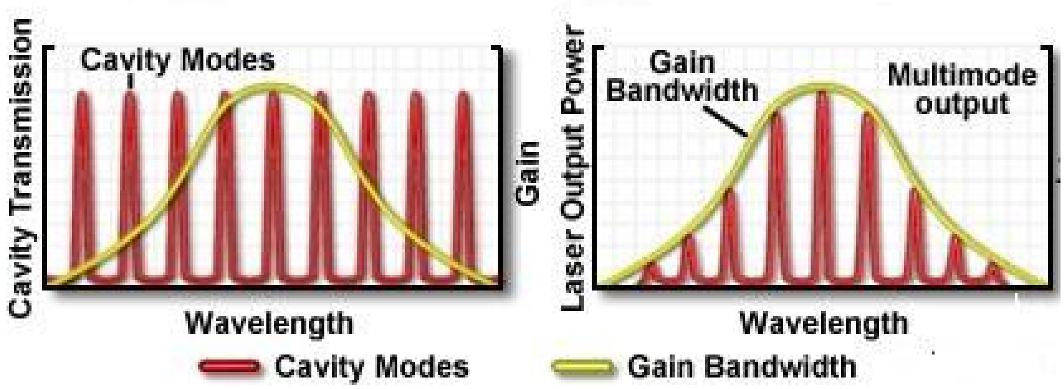

Figure 2.3: Multimode and single mode

Chapter 3
Materials and methods

3.1 Materials taken: Aluminum powder and graphite powder.

Note: Since the cadmium powder was not available in the laboratory, so we have used aluminum powder in the place of cadmium as a preliminary test for the function of microwaves. It was believed that this preliminary test will give an idea about the extent of coupling of microwaves already present in our laboratory. Note that graphite was kept under ambient.

3.2 Objective of the project: Cadmium carbide will be synthesized through microwave heating. A domestic microwave oven will be used. Dry and powdered starting materials will be taken. These will be used as received without further in-house purification. Solid state synthetic route will be adopted. Since, cadmium powder is not available in our laboratory it has been decided to perform few preliminary tests. Graphite powder (purity, company name) and aluminum metal powder were available. Since, thee materials i.e. graphite powder and aluminum powders were kept at ambient it was decided to study these raw starting materials using Infrared Spectroscopy. It was believed that presumably, these powders might have reacted with atmospheric oxygen and water. The oxide layers present at the surface of these powders may affect the microwave coupling capacity. In other words, the presence of an oxide layer on surface of these particles may alter the feasibility of the reaction. Hence the raw materials were characterized (see fig. 4.1, 4.2 and 4.3) using IR spectroscopy.

To test the behavior of graphite powder in microwaves a pinch of graphite was taken in a silica crucible (see fig.3.1).

Figure 3.1: Silica crucible 1 verses silica crucible 2

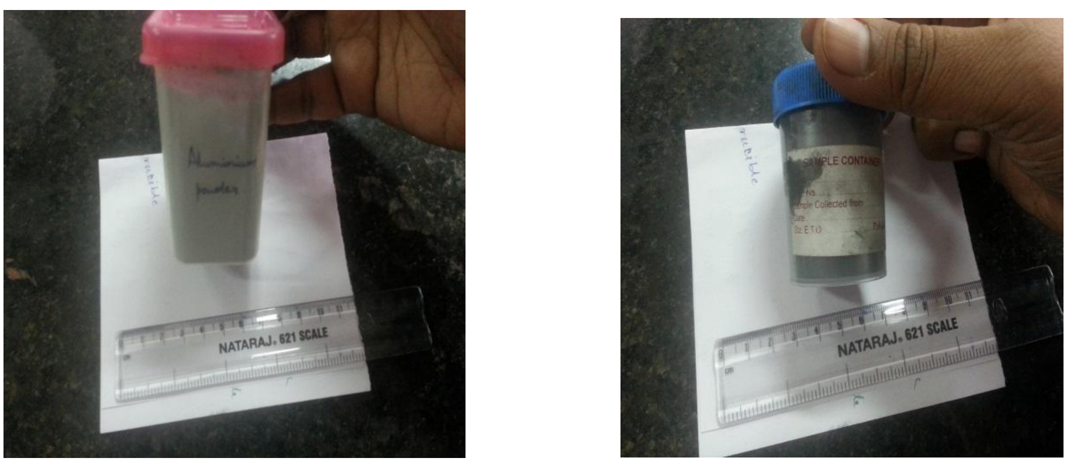

Figure 3.2: Aluminium powder and graphite powder

The dimensions of the silica crucible are given in Table 3.1.

Table: 3.1 Dimension of the silica crucible:

Dimension	Value (in mm)	
	For silica crucible 1	For silica crucible 2
Height	4.5 cm	2 cm
Diameter of the upper open part	7 cm	4 cm
Diameter of the lower close part	3.4 cm	2.7 cm
Wall thickness	2 mm	2 mm

The DMO was then switched on. The time and power used for exposure of microwaves are given in Table 3.

Table 3.2:List of microwave exposure of time and power.

Reactant/s taken	Power/Watt	Time
Graphite powder	900 W	20 sec.
Aluminum powder	900 W	3 min.
Mixture of aluminum powder and graphite powder	900 W	3 min.

Note that in the final report efforts will be made to synthesize cadmium carbide.

Samples were characterized using infrared spectroscopy (IR). The structure, function and procedure for making samples are discussed in the following section.

3.3 The infrared spectroscopy: Infrared spectroscopy is used to study the chemical properties of the compound and to identify the functional group of the compound. In IR spectroscopy the infrared radiations interact with the matter. IR spectroscopy is the absorption spectroscopy.

Equipment: FTIR (Fourier Transform Infrared) spectrophotometer is used for the IR spectroscopy.

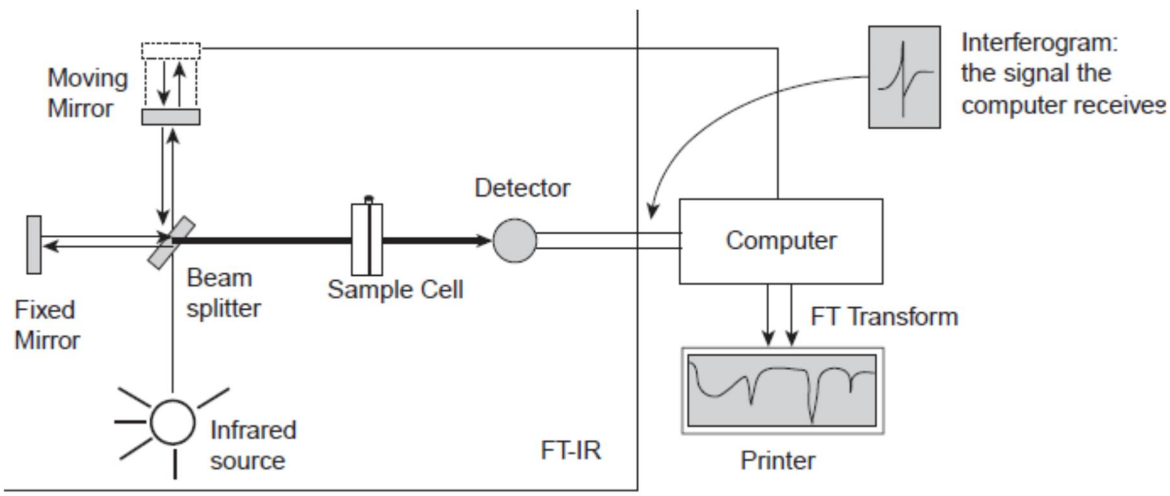

Figure 3.4: Fourier Transform Infrared spectrophotometer.

Since we know that there are two types of infrared spectrophotometer dispersive spectrophotometer and Fourier transform infrared spectrophotometer. Here we are using Fourier infrared spectrophotometer because it is more faster than the dispersive spectrophotometer.

Preparation of sample: IR spectroscopy is applicable for types of compounds whether it is solid, liquid or in gaseous form. Here we are using solid sample. For this we will inserted a KBr pellet in the instrument and keep the finely powdered sample into the KBr pellet. Press the sample under high pressure. Under pressure the Potassium bromides melts and seal the sample and we will obtained the spectra on the computer.

3.4 Working principle: It works on the principle of molecular vibrations. When the molecules are excited from lower energy level to higher energy level then they absorb infrared radiation. This is a quantized process. The molecule absorbs selected frequencies of infrared radiations [9]. The energy absorbed tends to increase the amplitude of the bonds in the molecules having vibrational frequencies.

3.5 Procedure:

(1) Domestic microwave oven should be attached with an electrical wiring system that has good earthing facilities.

(2) Graphite coupled with microwave at 900 W when exposed for about 20 seconds Sparks were seen but it never glowed. This suggests that the graphite may have contained some impurities that hindered coupling process.

(3) A pinch of aluminum powder was mixed with a pinch of graphite just to study whether microwave couples with aluminum- graphite mixture or not.

The reaction was performed under the following two conditions:

(1) The above said mixture i.e., mixture of aluminum powder and graphite powder was taken in a borosilicate glass test tube. The test tube was then kept in a borosilicate beaker (100ml). The mixture did not couple with microwaves.

(2) The above said reaction was repeated in a silica crucible (see figure 3.1). Sparks were observed. It is believed that microwaves coupled with the aluminum-silica mixture.

(3) The reaction mixture was heated at 900 W for 3 minutes. The domestic microwave oven was switched off for 10 minute to bring down the temperature of the domestic microwave oven cavity as a safety measure. The same mixture was again exposed to microwaves for 3 more minutes. Then we observed that there is no coupling between the mixtures. So there could be the formation of oxide layer on the surface of aluminum. That is why it has been not coupled.

Chapter 4

Results and discussions

Observation: When we exposure the graphite in the microwave oven then we observe spark which means that graphite is coupling with the microwaves.

The raw materials are investigated using infrared spectroscopy. A pinch of aluminum powder was mixed with KBr. It was then pressed to form a pellet. This pellet was subjected for IR-studies. The spectrum obtained is displayed in fig. 4.1.

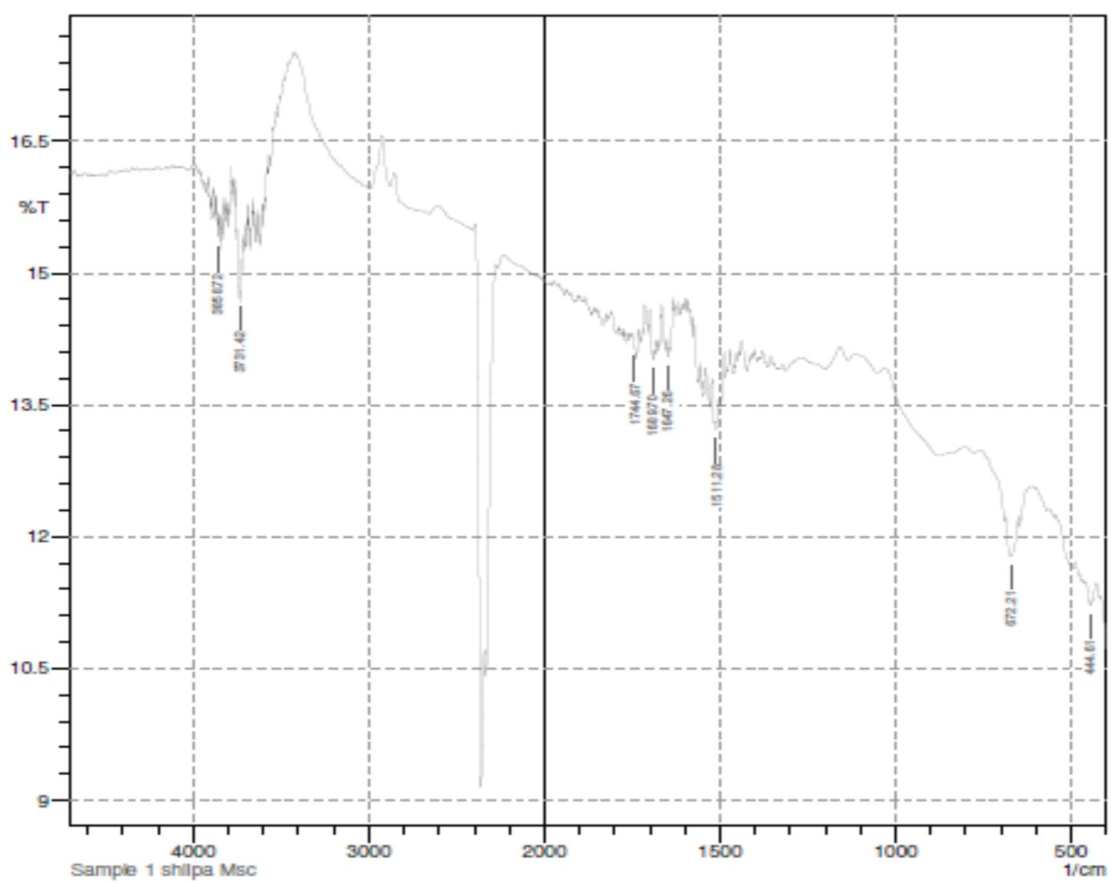

Fig. 4.1 IR spectrum of aluminium powder

The sample for graphite was also prepared in the same way. The obtained spectrum is shown in fig. 4.2.

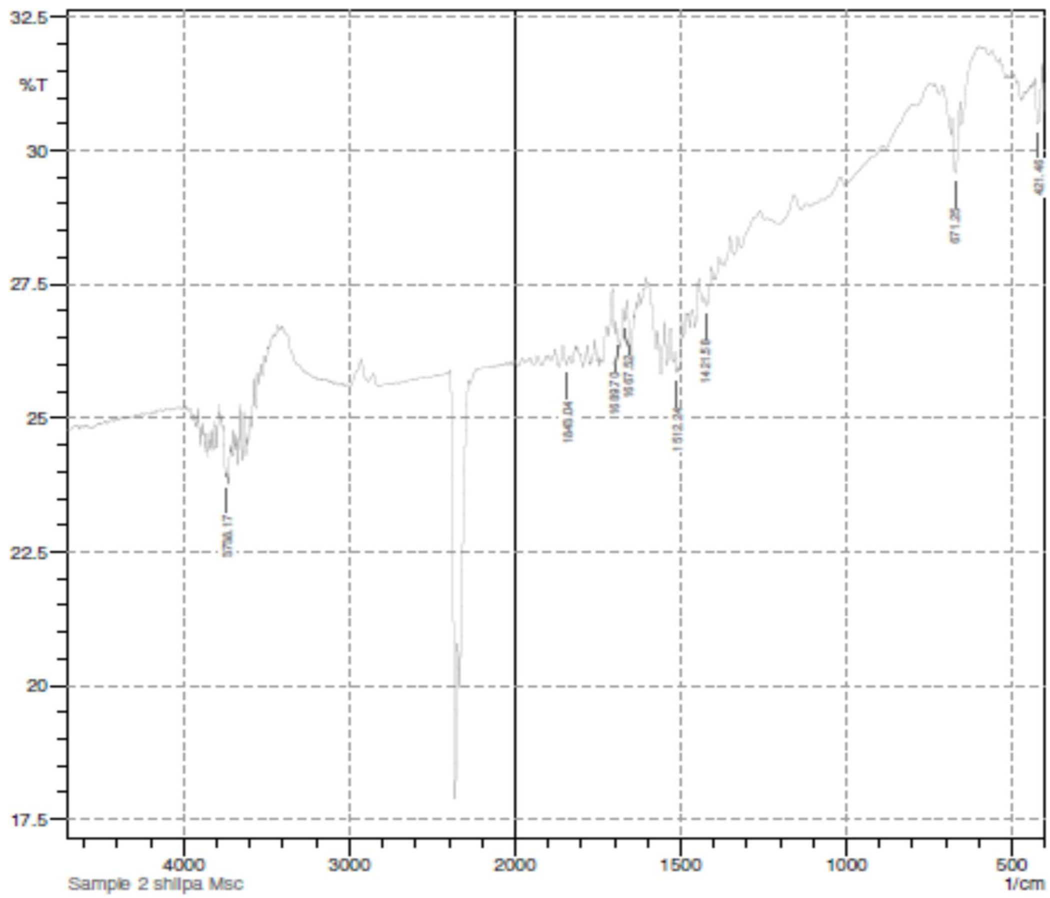

Figure 4.2: IR spectrum of graphite powder

The IR spectrum of aluminum-graphite mixture was also done by the same way as shown in figure.4.3

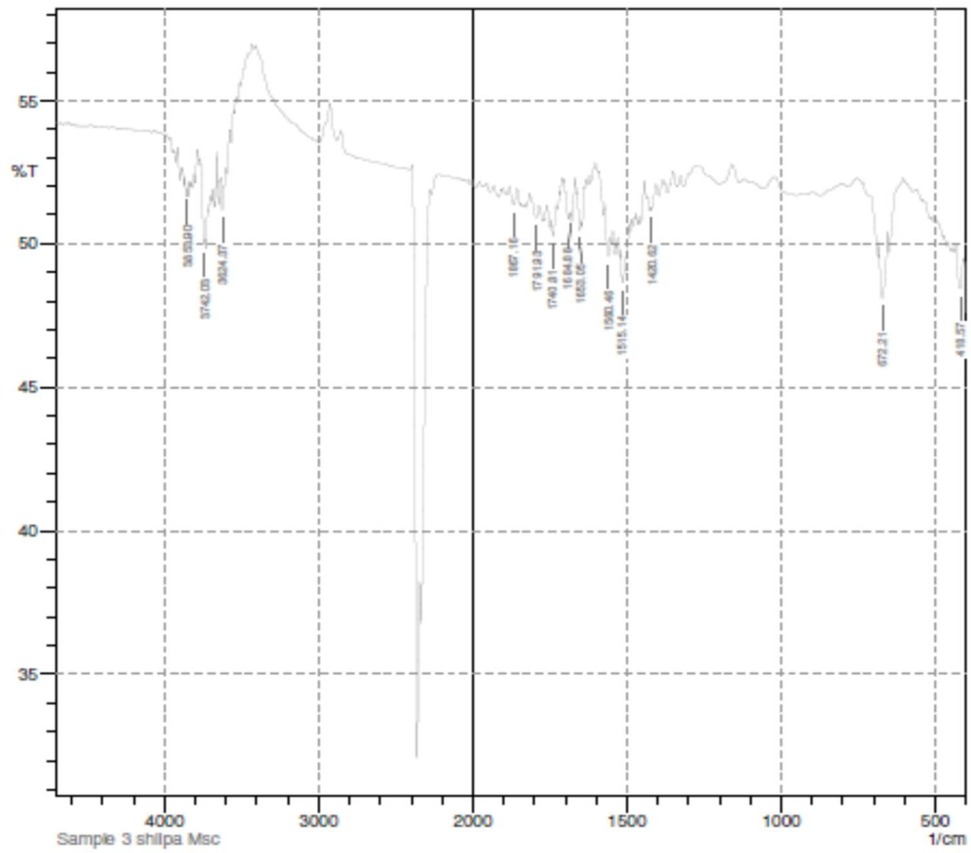

Fig 4.3: IR spectrum of aluminum-graphite mixture

Chapter 5

Future work

Efforts will be made to synthesize cadmium carbide through will be microwave heating. A domestic microwave oven will be used. Dry and powdered starting materials will be taken. These will be used as received without further in-house purification. Solid state synthetic route will be adopted. Graphite powder and cadmium metal powder will be mixed in molar ratios. The mixture will be transferred in a silica crucible. It will be then exposed to microwaves in a domestic microwave oven. Different power levels will be used. It was believed that this study will help us in understanding the role of conduction heating in carbides. Interfacial polarization may also play an important role in this. In case the reaction fails then it can be said that effect of conduction heating is not effective enough to carry out the reaction. The purpose of this study does not limit with formation or otherwise of the compound but effectiveness of microwave heating in laboratory and industrial purposes. The purity level of the starting materials also have important role in microwave heating.

The sample will be characterized using infrared spectroscopy.

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