STUDY ON CHARACTERIZATION OF OLIVINE MINERAL FROM SALEM AREA IN TAMILNADU

Submitted in partial fulfillment of the requirements

for the award of Bachelor of Science in Chemistry

Submitted by

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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of JAYAPRIYA S (40030013), SURIYA T (40030038) and SAMYUKTHA PARGAVI K (40030030) who carried out the project entitled **"STUDY ON CHARACTERIZATION OF OLIVINE MINERAL FROM SALEM AREA IN TAMILNADU"** under our supervision from December 2022 to April 2023.

Internal Guide Dr. K. CHENNAKESAVULU, Head of the Department Dr. J. KARTHIKEYAN.

Submitted for Viva voce Examination held on 08-05-2023

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DECLARATION

We JAYAPRIYA.S (40030013), SURIYA.T(40030038), and SAMYUKTHAPARGAVI.K(40030030) hereby declare that the Project Report entitled "STUDY ON CHARACTERIZATION OF OLIVINE MINERAL FROM SALEM AREA IN TAMILNADU" done by us under the guidance of **Dr. K. CHENNAKESAVULU**, Department of chemistry, Sathyabama institute of science and technology is submitted in partial fulfillment of the requirements for the award of Bachelor of science degree in Chemistry.

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CHAPTER 1

1.1INTRODUCTION:

Olivine is an important rock forming mineral group which belongs to silicate group specifically to the nesosilicate subclass. Composition of olivine are commonly expressed as molar percentages of forsterite and fayalite ex., $Fo_{70}Fa_{30}$. Olivine is a mixed crystal of Mg_2SiO_4 and Fe_2SiO_4, in which magnesium is dominant. it is mostly found in low silica mafic and ultramafic igneous rock in earth's upper mantle such as basalt, gabbro, dunite, diabase and peridotite. Norway is the main source of olivine where 50% of olivine is extracted for industrial usage. Most olivine is used as ornamental purpose are mined at San Carlos reservation in Arizona. The name of olivine derives from the unusual yellow-green to bottle-green color of the magnesium-iron olivine series. Gem-quality forsterite olivine is known as peridot. Olivine present in coastal region absorbs carbon dioxide in coastal region under some conditions which is a natural phenomenon. In our project we use characterization techniques to study the olivine composition, surface area of olivine in our sample.

1.2 Types of olivine mineral

Olivine contains seven different types of mineral group. The most common of which are forsterite and fayalite.

- Mg-rich forsterite Mg_2SiO_4
- Fe-rich fayalite Fe_2SiO_4
- Mn-rich Tephroite Mn_2SiO_4
- Mn-Fe rich knebelite $(Mn, Fe)_2SiO_4$
- Ca – rich larnite (Ca_2SiO_4)
- Ca-Mg rich Monticellite (Ca, Mg) SiO_4
- Ca-Fe rich kirschsteinite (Ca, Fe) SiO_4

1.3 Structure of olivine:

The structure of olivine was proposed by Bragg and brown. The structure of olivine can be described as a three-dimensional framework of silicon-oxygen tetrahedra, with magnesium or iron ions occupying interstitial sites within the framework. The chemical formula for olivine, $(Mg, Fe)_2SiO_4$, reflects the fact that magnesium (Mg) and iron (Fe) ions can substitute for each other in the crystal lattice. Each silicon (Si) atom is surrounded by four oxygen (O) atoms arranged in a tetrahedron, while each magnesium or iron atom is coordinated with six oxygen atoms in an octahedral arrangement. The tetrahedral and octahedral layers alternate to form a three-dimensional structure. The olivine structure is classified as orthorhombic, meaning it has three unequal axes perpendicular to each other. The unit cell of olivine is composed of eight silicon-oxygen tetrahedra and four magnesium or iron octahedra. Overall, the olivine structure is relatively simple and can be thought of as a framework of interconnected tetrahedra and octahedra. This structure gibes olivine its characteristic physical and chemical properties, including its hardness, density, and resistance to high temperature and pressures.

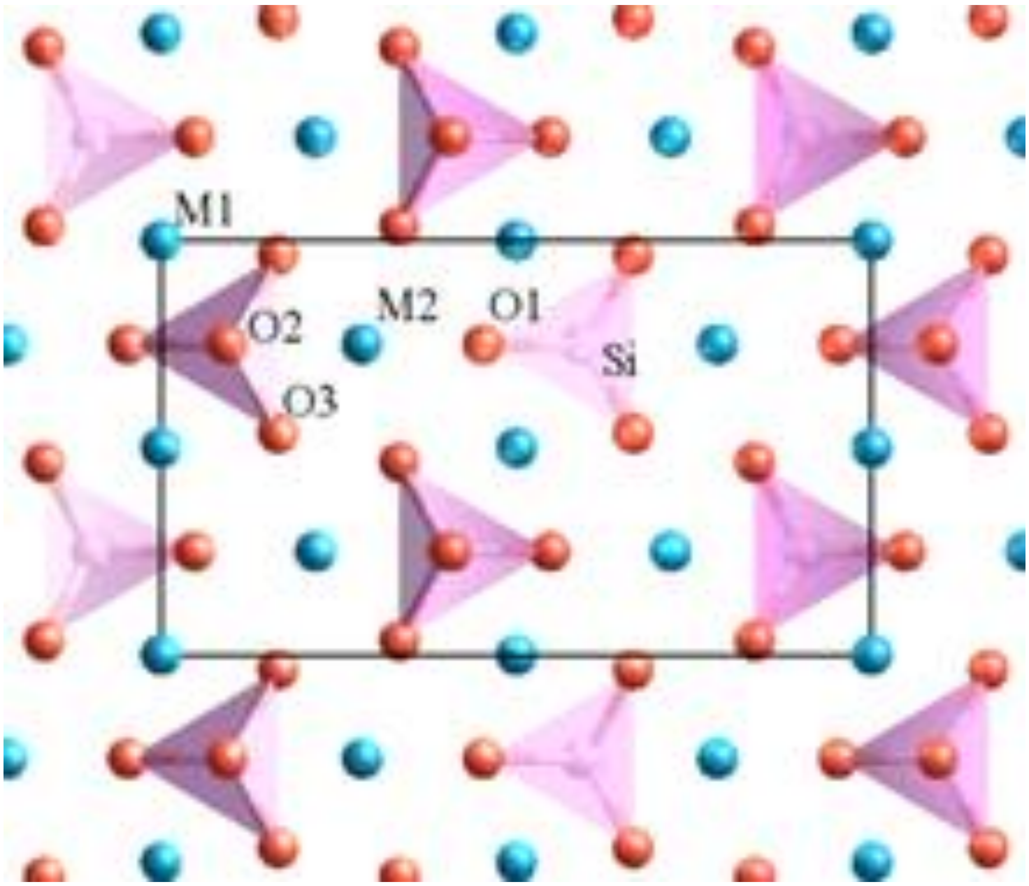

Figure 1

CHAPTER 2

LITERATURE AND REVIEW:

Characterization of olivine surface morphology and reactivity of by XRD, FT-IR, Surface area and PSD. This paper examines the surface morphology and reactivity of olivine using variety of analytical techniques including X-ray diffraction, FT-IR, Surface area and PSD.

In recent years, olivine has also attracted attention as a potential tool for mitigating climate change. When olivine reacts with carbon dioxide in the atmosphere, it can form magnesium carbonate and silica, which are stable compounds that do not contribute to greenhouse gas emissions. This has led to increased interest in the use of olivine as geoengineering tool to sequester carbon dioxide from the atmosphere.

Physical properties:

Olivine is relatively hard and resistant to scratching, with a hardness of 6.5-7 on the Mohs scale. This property makes olivine useful in industrial applications where abrasion resistance is important, such as in sandblasting and grinding operations. Specific gravity of olivine is around 3.2 to 4.5. It has glassy luster and a conchoidal fracture. Olivine is also notable for its high melting point, which makes it a common mineral in basaltic and ultramafic rock. It has high melting point around 1,780 to 1,930 degrees Celsius and resistance to chemical reagents; olivine is an important refractory material.

Chemical properties:

Solid solution: Olivine is a solid solution of magnesium (Mg) and iron (Fe) ions within the silicate framework. The relative amounts of Mg and Fe in olivine can vary, depending on the specific composition of the rock in which it is found.

Isomorphism: Olivine exhibits isomorphism, which is the ability of minerals with similar structures to substitute for one another in crystal lattices. This means that olivine can substitute for other minerals in certain rock formations, and vice versa.

Chemical reactivity: Olivine is relatively unreactive chemically, but it can undergo weathering and alteration over time. This can result in the formation of secondary mineral such as serpentine, which is an important mineral in the formation of certain types of metamorphic rocks.

CHAPTER 3

AIM AND SCOPE:

Olivine characterization aim is to study the physical and chemical properties of olivine such as chemical composition, crystal structure and surface area. It has an important application in various fields, including geology, material science and environmental science. Olivine has various scopes in industrial uses as an abrasive, super green fuel, removal of arsenic from water, agriculture, olivine act as positive electrode for Li-ion batteries, olivine has the potential to sequester carbon dioxide from the atmosphere and has applications in environmental remediation. Characterization techniques can be used to study its effectiveness in these applications. Overall, olivine characterization aims to understand the properties and behavior of this important mineral and its applications on various fields.

CHAPTER 4

METHODS AND METHODOLOGY:

Our crystal sample is powdered using Pulverizer and the powdered form is again grinded manually to obtain fine powder of olivine for the characterization.

Figure 2: Sample

Figure 3: Pulverizer

We used characterization methods such as XRD, FT-Raman, surface area, particle size distribution to study chemical composition, crystal structure and surface area of olivine.

X-RAY DIFFRACTION (XRD): XRD is a common technique used to determine the crystal structure and composition of minerals. In olivine, XRD can be used to determine its crystal structure and composition.

- XRD is based on constructive interference of monochromatic X-rays and a crystalline sample. These X-rays are generated by a cathode ray tube, filtered to produce monochromatic radiation, collimated to concentrate and directed towards the sample. The interaction of incident rays with the sample produces constructive interference when conditions satisfy Bragg's law.
- XRD is a nondestructive technique that provides detailed information about the crystallographic structure, chemical composition and physical properties of a mineral. X-rays are shorter wavelength electromagnetic radiation that are generated when electrically charged particles with sufficient energy are decelerated. The intensity of the diffracted rays scattered at different angles of material are plotted to display a diffraction pattern.

Figure 4

RAMAN SPECTROSCOPY: Raman spectroscopy is a technique used to study the vibrational modes of molecules. In olivine, Raman spectroscopy can be used to identify the mineral and determine its crystal structure.

- Samples of single domain olivine were mounted onto slides with epoxy and polished prior to use in the Raman setup displayed. Raman shift was observed.

- Intensity ratios of the changing vibrational energies were extrapolated from the data collected by integrating spectral peals such as those in figure 4: the ratios were normalized to the largest peak and plotted against the angle of rotation.
- Raman spectroscopy is much faster than XRD for examining crystal orientation.
- The Raman spectrum of olivine varies systematically with the orientation angle of the sample. The ratio of intensities of the Raman peaks can be used to help determine the absolute orientation of the crystal axis in the sample.

Figure 5

BET (BRUNAUER-EMMETT-TELLER): BET analysis is a commonly used technique for studying the surface area and porosity of materials, including minerals such as olivine. In olivine, BET analysis can be used to determine the surface area of the mineral by measuring the absorption and desorption of gas molecules on the surface of the material. The aim of a BET analysis study is to provide quantitative information about the surface area, pore size distribution, and pore volume of a material.

In a BET analysis study of olivine, a sample of the mineral would typically be subjected to a series of treatments and measurements, including:

1. Sample preparation: The olivine sample would need to be cleaned and dried to remove any surface contaminants or water molecules that could interfere with the analysis.

2. Nitrogen adsorption: The olivine sample would need to be exposed to nitrogen gas at low temperatures and pressures. The nitrogen molecules would adsorb onto the surface of the olivine, filling any available pore spaces.
3. BET equation: The data from the nitrogen adsorption experiment would then be analyzed to the surface area of the olivine.
4. Pore size distribution: By analyzing the shape of the nitrogen adsorption isotherm, the pore size distribution of the olivine sample can be determined. This information can be used to determine the types of pores present in the olivine (micropores, mesopores or macropores) and the size range of each type.
5. Pore volume: The total volume of the olivine sample can also be calculated from the nitrogen adsorption data. This information can be useful in predicting how the olivine will behave in various industrial or geological applications.

Overall, a BET analysis study of olivine can provide important information about the surface properties and porosity of the mineral. Which can be used to better understand its behavior in a wide range of settings.

CHAPTER 5

RESULT AND DISCUSSION:

5.1 RAMAN SPECTRA:

The majority of the peaks, specifically 822 and 856, correspond to the symmetric stretching of olivine. This is consistent with previous studies that have used Raman spectroscopy to analyze olivine samples. The peak positions fall within the range of 847 to 857, which suggests that the olivine present in the sample is likely to be of good quality. Another peak observed in the data is at position 678, which corresponds to the bending vibration of magnesium oxide (MgO). This peak is indicative of the presence of MgO in the sample.

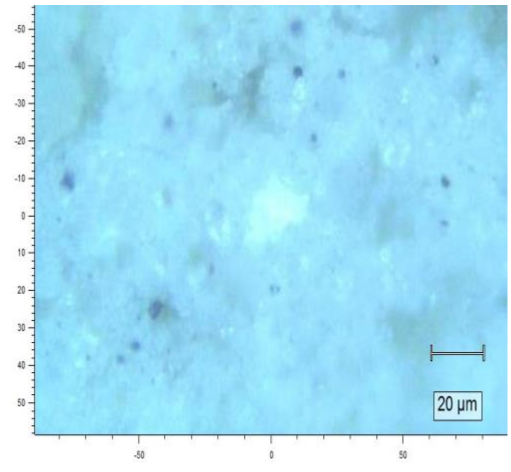

Figure 6

RAMAN GRAPH:

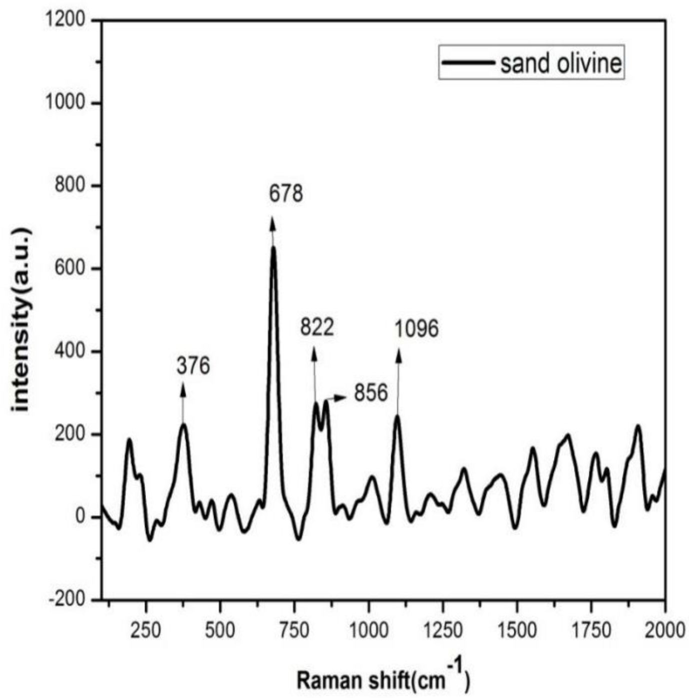

Figure 7

A peak at position 1096 corresponds to the stretching bond of silicon and oxygen (Si-O). This peak indicates the presence of Si-O bonds in the sample, which is commonly found in silicate minerals. Lastly, a peak at position 376 corresponds to the presence of FeO_2. This peak suggests the presence of iron oxide (FeO_2) in the sample, which is a common component of many minerals. The FT-RAMAN data suggests that the sample analyzed contains olivine, magnesium oxide, silicate minerals, and iron oxide. The information obtained from this analysis can help in identifying and characterizing the mineral composition of the sample.

5.2 XRD

The XRD peaks observed at 31 and 36 degrees 2θ (theta) in the XRD pattern are characteristic of the forsterite group of olivine minerals. Forsterite is a magnesium-rich member of the olivine mineral group with the chemical formula Mg_2SiO_4. The forsterite group also includes other magnesium-rich members such as fayalite (Fe_2SiO_4) and tephroite (Mn_2SiO_4).

In X-ray diffraction (XRD) analysis, the diffraction pattern is obtained by exposing a powdered sample to X-rays and measuring the angle and intensity of the diffracted X-rays. The angle at which the diffraction peaks occur is related to the interatomic spacing of the crystal lattice of the sample. The intensity of the peaks is related to the number of atoms and their arrangement within the crystal lattice.

In the case of olivine minerals, the forsterite group has a characteristic XRD pattern with peaks at 31 and 36 degrees 2θ, which correspond to the (020) and (113) planes of the forsterite crystal lattice. These peaks are sharp and intense, indicating a well-ordered crystal structure with a high degree of crystallinity.

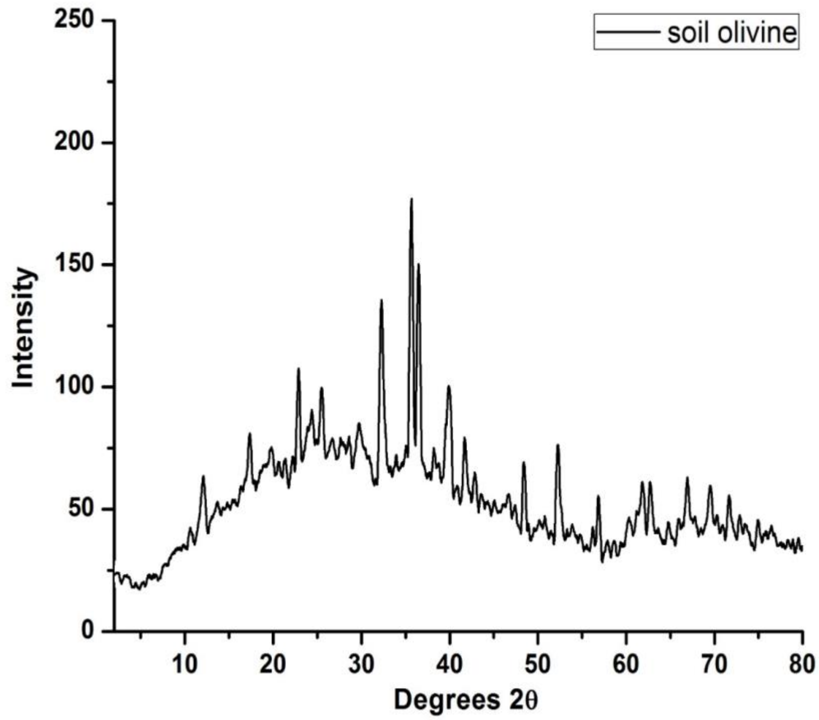

Figure 8

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The identification of forsterite in a sample using XRD analysis can be useful in a variety of applications, such as mineral identification, geological studies, and materials science. Forsterite has unique physical and chemical properties, such as high thermal stability, high melting point, and low thermal expansion coefficient, which make it suitable for use in a variety of applications such as refractories, ceramics, and high-temperature insulation.

5.3 SURFACE AREA (BET):

The BET surface area of the olivine powder sample was determined to be 16.466 m^2/gm, with a pore volume of 0.0082 cm^3/gm and a pore diameter of 2 nm. To determine the BET surface area, the Brunauer-Emmett-Teller (BET) method was used. First, a known amount of the sample was degassed under vacuum to remove any adsorbed gas or moisture. The sample was then exposed to a series of different partial pressures of a gas adsorbate, such as nitrogen, at different temperatures. The amount of gas adsorbed at each pressure was measured using a gas adsorption instrument, such as a surface area analyzer.

The data obtained from the adsorption isotherm was then analysed using the BET equation, which relates the amount of adsorbed gas to the surface area of the sample. The BET surface area was calculated by plotting the quantity $(P/P_o) / (V(1-V))$ against the relative pressure (P/P_o), where V is the volume of gas adsorbed and P_o is the vapor pressure of the gas at saturation. The slope of the resulting linear plot was used to calculate the BET surface area.

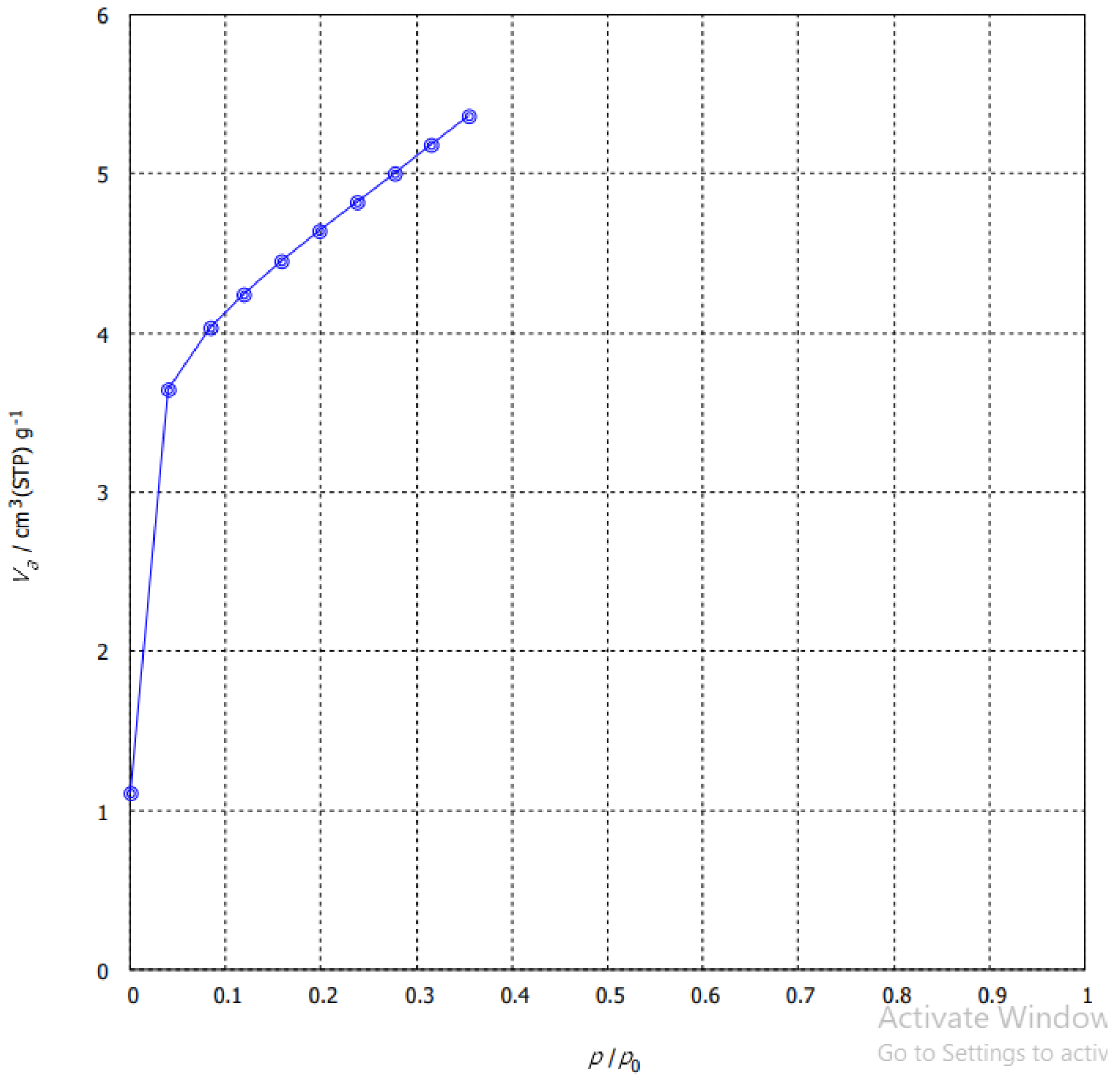

Figure 9

To determine the pore volume and diameter, the sample was analysed using the Barrett-Joyner-Halenda (BJH) method. This involves calculating the pore size distribution from the desorption branch of the isotherm using the Kelvin equation. The BJH method assumes that the pores are cylindrical and non-interconnected. The pore volume was calculated by integrating the pore size distribution curve, and the average pore diameter was calculated from the maximum of the cumulative pore volume versus pore diameter curve.

5.4 PARTICLE SIZE DISTRIBUTION

There are several methods available to measure the particle size distribution of olivine powder, including sedimentation, laser diffraction, and microscopy. One of the commonly used methods is laser diffraction. The following is a brief description of the laser diffraction method:

Dispersion of the powder sample: A small amount of olivine powder is dispersed in a liquid medium to ensure uniformity of the sample. This is typically done using an ultrasonic bath.

Measurement: The dispersed sample is then subjected to laser light. The particles in the sample scatter the light, and the scattered light is captured by a detector. The angle of scattering is proportional to the size of the particles, and the intensity of scattered light is proportional to the number of particles.

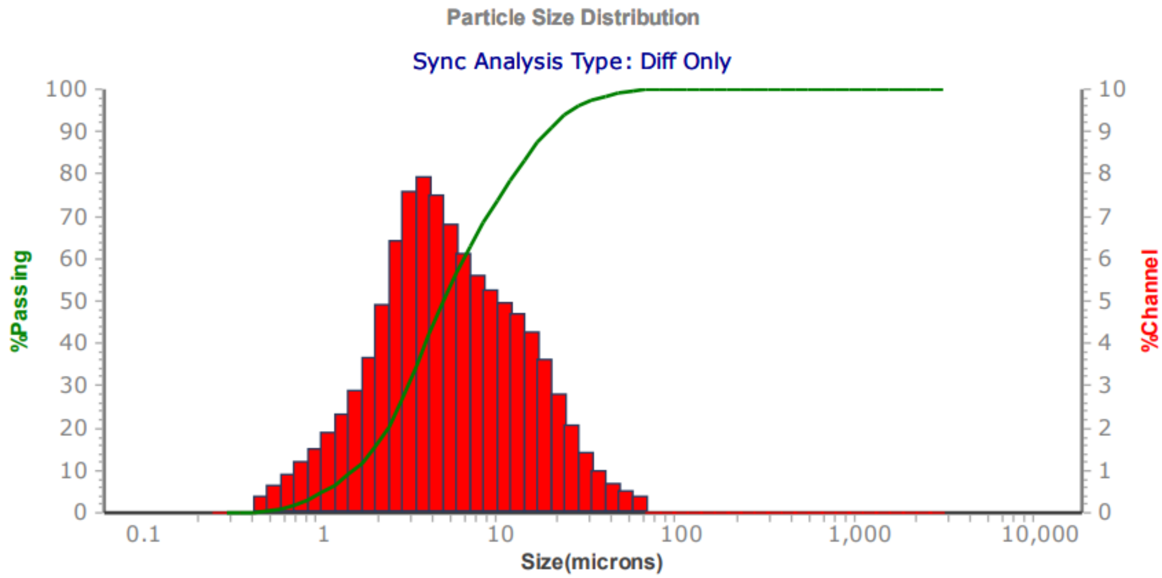

Figure 10

Analysis: The detector output is analyzed by software, which converts the scattering pattern into a size distribution graph. The graph shows the number of particles in each size range.

The particle size distribution of the olivine powder can be expressed as the mean particle size or the median particle size. The result of 7.6 microns likely represents either the mean or median particle size obtained from the laser diffraction analysis. It is important to note that the particle size distribution can vary depending on the method used and the conditions of the analysis.

CHAPTER 6

CONCLUSION:

- The FTRAMAN peaks at 822 and 856 indicate olivine's symmetric stretching, consistent with previous studies. Peak positions of 847 to 857 suggest good quality olivine. Peaks at 678, 1096, and 376 correspond to MgO, Si-O bonds, and FeO_2, respectively, indicating the sample contains olivine, magnesium oxide, silicate minerals, and iron oxide.
- The XRD pattern at 31 and 36^o 2θ indicates the presence of forsterite, a magnesium-rich member of the olivine mineral group. XRD measures crystal lattice spacing and atom arrangement, showing the well-ordered forsterite structure. Forsterite has unique properties, making it useful in various applications, including refractories and ceramics.
- The Brunauer-Emmett-Teller method was used to determine the BET surface area. The BET surface area of the olivine powder sample is 16.466 m^2/gm. Pore volume is 0.0082 cm^3/gm with a pore diameter of 2 nm.
- Olivine particle size is 7.6 microns, obtained by laser diffraction analysis.

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