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Effect of Laser Induced Potassium Oxalate ($C_2K_2O_4$) Nanopowder

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ABSTRACT

In material science the word characterization designates to the broad and general process by which a materials structure and properties are enhanced and measured. It is a fundamental process in the field of material science. The advent of new technologies helps to improve the quality of the research by understanding the different behaviors and properties of the materials. In this work we investigated the nanopowder potassium oxalate and its interaction with He-Ne laser are studied. Potassium oxalate is a strong dicarboxylic acid occurring in many plants and vegetables. It is produced in the body by metabolism of glyoxylic acid or ascorbic acid. It is not metabolized but excreted in the urine. It is used as an analytical reagent and general reducing agent. It is an odorless white solid. Now in this study we report the influence of laser heat treatment of nanopowder potassium oxalate. The laser treated potassium oxalate samples are characterized and analyzed by X-ray diffraction, UV-visible absorption, FTIR spectra and SEM. From the X-ray analysis we observed that the laser treated potassium oxalate with the structure of orthorhombic phase structure and the crystalline size in the range of 53 nm to 59 nm. The UV-Visible spectrum also confirms the presence of potassium oxalate nanoparticles with the average band gap energy 5.3 -5.7 eV. FTIR confirms the presence of potassium oxalate nanoparticles with various groups. The average particle size observed in both SEM and XRD measurements are nearly equal values.

1. Introduction

Nanoparticles are gaining importance because of their applications in various fields as well as their considerable stability. Potassium oxalate is a strong dicarboxylic acid. It is also called as dipotassium oxalate, potassium neutral oxalate, ethanedioic acid, dipotassium salt. It is odorless and white crystals, used chiefly as a bleaching agent and in medical tests as an anticoagulant. It is used as a reagent in analytical chemistry, source of oxalic acid, removing stains and it is used as a developer in the platino type process of photography. Sinks in and mixes slowly with water [1-3]. As with other alkali metals, potassium is highly reactive to water with the evolution of hydrogen because of it reacts violently with water. it only occurs in nature as ionic salts. Potassium has a silvery gray metallic appearance, but its compounds are more frequently used in industrial and chemical applications. It is a chemical element, atomic number 19, atomic weight 39.102, symbol K. With the use of laser heat treatment on semi conducting materials gives necessary enhancement either in conductivity or in band gap energy with the use of proper laser parameters [4-7].

This paper investigation the effect of laser irradiation on potassium oxalate nanoparticle with different time exposures. The structural analysis of the samples is analyzed using X-ray diffract meters. Similarly, the investigations such as UV and FTIR are also used for with and without laser irradiated samples. SEM studies show interesting information pertaining to band gap energy and particle size.

2. Experimental Methods

Commercially available powder $C_2K_2O_4$ is used for investigation. Laser treatments are carried out by using 5.0 mw low power He-Ne gas laser with red light of (wavelength 633 nm) (Fig. 1) [8, 9]. The structural analysis of with and without laser irradiated samples are analyzed using analytical X-ray diffractometers (copper Kα radiation of wavelength λ as 1.54060 Å and 1.54443 Å). This system recorded the intensity as a function of Bragg's angle. While the average crystalline size of the particles can be estimated using full width at half maximum (FWHM) value of the X-ray diffraction peaks. The optical investigation of the samples such as UV and FTIR studies are performed using Perkin Elmer UV/VIS spectrophotometer (λ 365) and Perkin Elmer FTIR spectrometer. $C_2K_2O_4$ nanoparticles are analyzed using SEM (ZEISS SEM instrument) micrographs. The laser processing parameters used for laser irradiation of samples are given in Table1 and the properties of sample used is given in Table 2.

Table 1 Time duration of laser irradiation for sample with different power of laser

Sample Number	Weight (g)	Time Duration of Laser Irradiation (sec)
1	0.5	Without laser irradiation
2	0.5	15 minutes (with He-Ne laser irradiation)
3	0.5	30 minutes (with He-Ne laser irradiation)

Table 2 Properties of $C_2K_2O_4$

Parameters	Values
Chemical formula	$C_2K_2O_4$
Molecular formula	$K_2C_2O_4$
Molecular weight	166.215 g/mol
Appearance	Odourless white crystals
Solubility in water	330 g/L
Density	2.13 g/cm^3
Melting point	356 °C

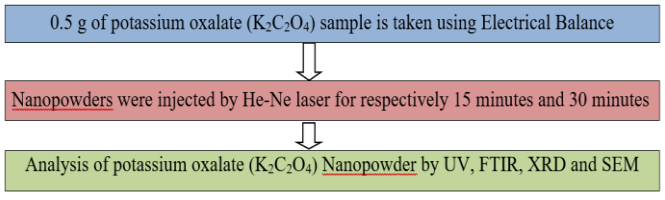

Fig. 1 Experimental flowchart

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3. Results and Discussion

3.1 UV-Vis Studies

UV spectroscopy is a type of absorption spectroscopy in which light of ultraviolet range (200-400 nm) is absorbed by the molecules. Absorption of the ultraviolet radiation results in the excitation of the electrons from the ground state to higher state. The energy of the ultra violet radiation that are absorbed is equal to the energy difference between the ground state and higher energy states [10]. Energy of the nanopowder with and without laser irradiated samples can be evaluated from the absorption spectra, optical constant and band gap energy. The optical band gap energy were evaluated from the absorption spectra, which related to the large band gap of about (5.3 eV - 5.7 eV) (Figs. 2-4 and Table 3).

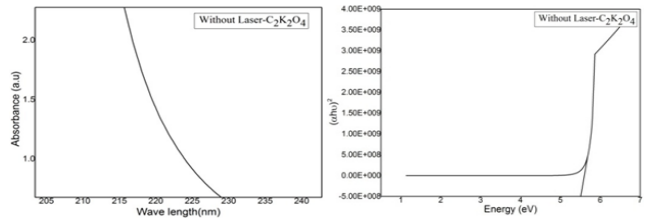

Fig. 2 (a) Absorbance spectra of $C_2K_2O_4$ (without laser) and (b) Transmittance spectra of $C_2K_2O_4$ (without laser)

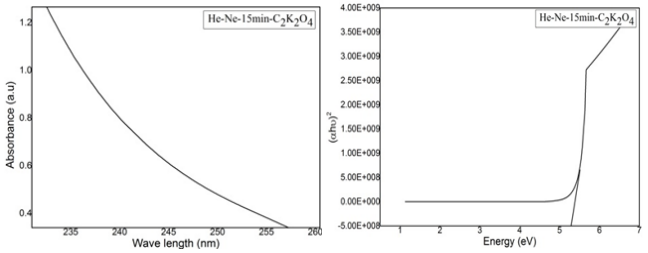

Fig. 3 (a) Absorbance spectra of $C_2K_2O_4$ (He-NeLaser-15 min) and (b) Transmittance spectra of $C_2K_2O_4$ (He-NeLaser-15 min)

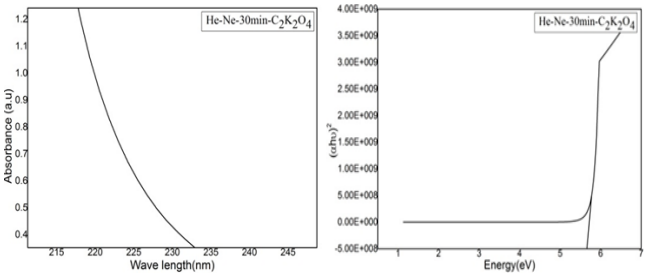

Fig. 4 (a) Absorbance spectra of $C_2K_2O_4$ (He-NeLaser-30 min) and (b) Transmittance spectra of $C_2K_2O_4$ (He-NeLaser-30 min)

Table 3 The band gap energy of each sample

Sample	Time duration of laser irradiation (s)	Band gap energy (eV)
1.	Without laser irradiation	5.3
2.	15 minutes (with He-Ne laser irradiation)	5.4
3.	30 minutes (with He-Ne laser irradiation)	5.7

3.2 FTIR Studies

The FTIR Spectrum of $C_2K_2O_4$ nanoparticles are given in Figs. 5-7. The function analysis from the FTIR data is given in Tables 4-6.

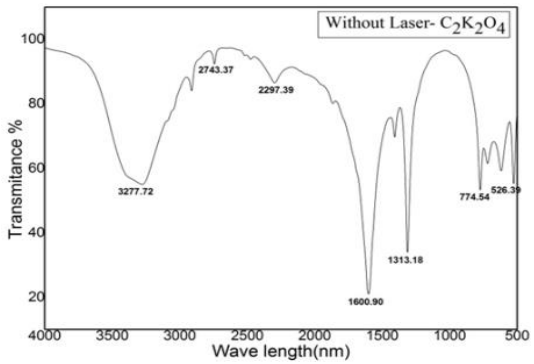

Fig. 5 FTIR without laser treated $C_2K_2O_4$

Table 4 FTIR functional analysis without Laser-$C_2K_2O_4$

Wave	Group	Vibration	Intensity
3277	O-H	Stretching	Strong
2743	C-H	Stretching	Medium
2297	C≡C	Stretching	Variable
1600	C=C	Stretching	Medium-weak
1313	O-C	Stretching	Medium-Strong
774	C-H	Bending	Strong-medium
526	C-X	Bending	Medium-weak

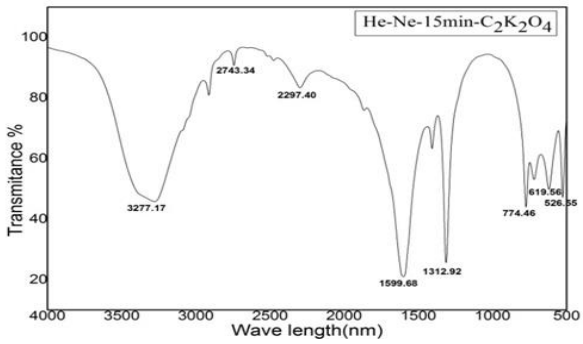

Fig. 6 He-Ne laser treated 15 min-$C_2K_2O_4$

Table 5 FTIR functional analysis (He-Ne-15min-$C_2K_2O_4$)

Wave	Group	Vibration	Intensity
3277	O-H	Stretching	Strong
2743	C-H	Stretching	Medium
2297	C≡C	Stretching	Variable
1599	C=C	Stretching	Medium-weak
1312	O-C	Stretching	Medium-Strong
774	C-H	Bending	Strong-medium
619	C-H	Bending	Strong
526	C-X	Bromoalkanes	Medium-weak

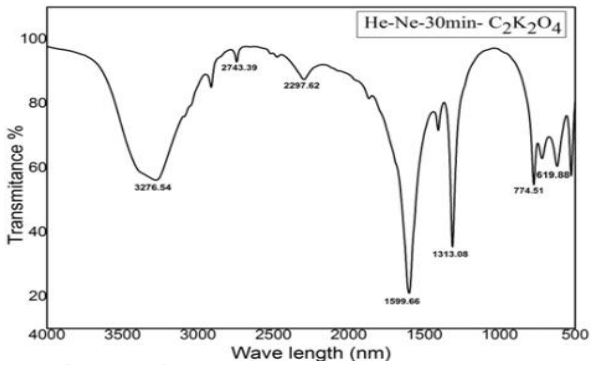

Fig. 7 He-Ne laser treated 30 min-$C_2K_2O_4$

Table 6 FTIR functional analysis (He-Ne-30min-$C_2K_2O_4$)

Wave	Group	Vibration	Intensity
3276	O-H	Stretching	Strong
2743	C-H	Stretching	Medium
2297	C≡C	Stretching	Variable
1599	C=C	Stretching	Medium-weak
1313	O-C	Stretching	Medium-Strong
774	C-H	Bending	Strong-medium
619	C-H	Bending	Strong

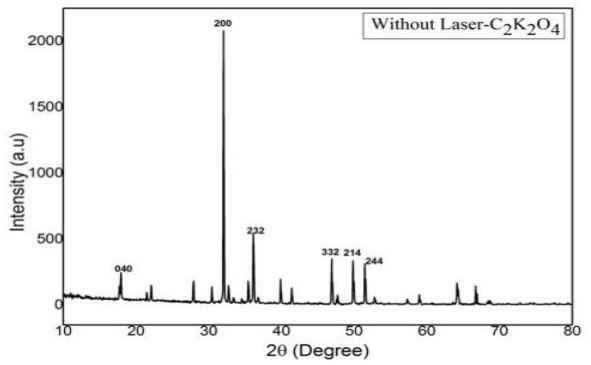

Fig. 8 XRD spectrum analysis of without laser treated $C_2K_2O_4$ nanoparticles

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3.3 XRD Analysis

The X-ray diffraction pattern of with and without laser treated samples are shown in Figs. 8 and 9; and Tables 7-10. The powder X-ray diffraction studies were performed at Alagappa University Science Center in Karaikudi. A beam voltage of 40 kV and a beam current 30 mA were used. The data were collected in the 2θ range (10-80) with continuous scan speed of 0.2 deg/min. The average particle size (D) was determined using the Scherrer's equation, D= Kλ / βcosθ, where D is the crystallite size, K is the shape factor, being equal to 0.9, λ is the X-ray wavelength, β is the full width at half maximum of the diffraction peak, and θ is the Bragg diffraction angle in degree.

Table 7 Structural parameters of $C_2K_2O_4$ nanoparticles without laser irradiation

S.No	Peak Position	hkl value	D spacing	FWHM	Particle Size(nm)
1	17.9403	040	4.94446	0.1476	54.51
2	32.7484	200	2.73470	0.1476	56.13
3	36.8207	232	2.44106	0.1476	56.76
4	47.7741	332	1.90386	0.1476	58.91
5	51.5025	214	1.77445	0.2460	35.87
6	52.8372	244	1.73273	0.1476	60.20

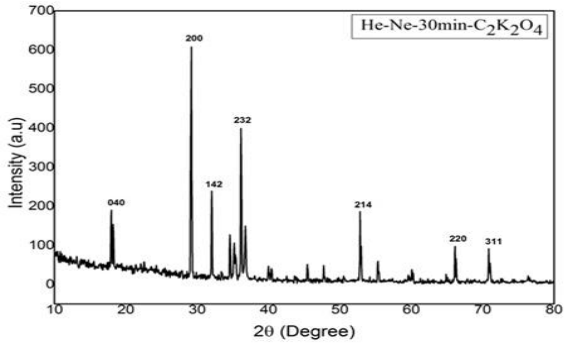

Fig. 9 XRD spectrum analysis of He-Ne laser Treated 30 min $C_2K_2O_4$ nanoparticles

Table 8 Parameters of $C_2K_2O_4$ nanoparticles He-Ne laser treated with 30mins

S.No	Peak Position	hkl value	D spacing	FWHM	Crystallite size (nm)
1	18.2478	040	4.94210	0.1476	54.54
2	29.1579	200	3.06275	0.1476	55.64
3	32.0328	142	2.79413	0.1476	56.02
4	36.7416	232	2.44613	0.1476	56.74
5	52.8115	214	1.73351	0.1476	60.12
6	66.0713	220	1.41415	0.1476	64.24
7	70.8315	311	1.33034	0.1476	66.07

Table 9 crystallite grain size of $C_2K_2O_4$ samples

S.No.	Samples	Grain size in nm
1	Without laser $C_2K_2O_4$	53.73
2	He-Ne laser 30min $C_2K_2O_4$	59.05

Table 10 The comparative study of particle size of potassium oxalate nanoparticles

S. No.	Sample	XRD Crystallite (nm)	SEM Particle (nm)
1	Without laser irradiation	53	20-127
2	30 minutes (with He-Ne laser irradiation)	59	31.47 – 87.4

3.4 SEM Analysis

The SEM Analysis of with and without laser treated samples are shown in Figs. 10 and 11. From the SEM analysis of potassium oxalate nanoparticle, it is observed that the particles are in the orthorhombic structure within the particle size are in the range about 20 - 127 nm (sample 1) and 31.47 – 87.40 nm (sample 3).

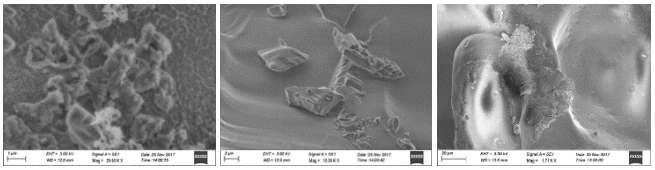

Fig. 10 SEM analysis of He-Ne laser treated 30 min $C_2K_2O_4$ nanoparticles

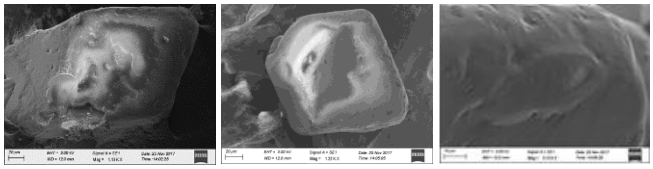

Fig. 11 SEM analysis of non-laser treated $C_2K_2O_4$ nanoparticles

4. Conclusion

This present work investigated and reported the effect of laser He-Ne induced nanoparticles of potassium oxalate. From the UV spectrum studies, we observed variation in band gap energy for various laser treated samples (values vary from 5.3 – 5.7 eV). The different peaks observed from FTIR spectrum with respect to each functional group for the synthesised samples. From the X-ray diffraction studies, we observed the structure of potassium nanoparticle is orthorhombic phase structure. The crystallite grain size of the samples was calculated as 53 nm (without laser) and 59 nm (He-Ne laser 30 mins). From the SEM analysis of potassium oxalate ($C_2K_2O_4$) nanoparticles, we observed that the particles are in the orthorhombic phase structure. The particle sizes are in the range about 20-127 nm (without laser) and 31.47-87.40 nm (He-Ne laser 30 mins). In general, when the laser power with increased with time exposure gives improved result in band gap energy as well as in grain size structure. The average particle size observed in both SEM and XRD measurements are nearly equal value.

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About the Conference…

National Conference on "Nano National Summit: Current Trends and Future Perspectives – NNSCTFP-2019" has been convened by Dr. A. Leema Rose, Associate Professor, PG and Research Department of Chemistry, Holy Cross College (Autonomous), Tiruchirappalli, India, at her designated venue on 9th & 10th January 2019.

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