

Distribution of Microcrystalline Quartz in Glassy Fulgurites from Garuamukh and Kimin, India

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Abstract

The presence of microcrystalline quartz particles in fulgurites of Garuamukh and Kimin has been investigated. The compositional and structural studies were carried out at room temperature by using X-ray fluorescence (XRF), X-ray diffraction (XRD) and Fourier transform infrared (FTIR) spectroscopic techniques. The compositional result shows the major constituent of the fulgurites to be SiO₂ with miniature quantities of oxides of Al, Ti, Fe, Ca, and Na. The X-ray diffraction quantified the mixtures of identical amorphous and micro-crystalline phases in the fulgurites samples. Systematic investigations of microcrystalline quartz particles have been carried out in mid-infrared region of 500 - 1000 cm⁻¹ based on the assignment of infrared bands of the structural group SiO₄ tetrahedra. In infrared Si-O-Si bending vibration region of quartz, the octahedral characteristic peak is explored in the fulgurites samples with the variation of the particle sizes which is indicative to the presence of microcrystalline quartz. The distribution of the microcrystalline quartz is ascertained by calculating the extinction coefficient. The present study has significant implications in understanding the thermodynamic properties of naturally occurring glasses, which are formed by shock metamorphism.

Keywords

Fulgurites, Microcrystalline Quartz, Spectroscopic Method

1. Introduction

Numbers of phenomena are responsible for making natural glass on Earth including meteorite impact, lightning

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and volcanic activity. The glassy object made as a result of a cloud-to-ground lightning discharge is called fulgurites. A typical lightning strike may disperse up to 1 GJ per flash and a small fraction of it (~1 MJ) reaches the ground [1]. It is accompanied by temperatures momentarily reaching between 18,000 and 39,000 K depending on moisture content in the air and this temperature is tremendously higher than the melting point of silica [1] [2]. This energy raises the temperature and pressure of the ground mass (soil, sand or rock) in the lightning channel accelerating material in and around the channel radially outward. It results in shock metamorphic effects. The siliceous components of the sand, soil, or rock fused to form solidified glass and a fragile hollow tube with a diameter of a few centimeters and a length that can vary from centimeters to meters [3] [4]. The outer surface of fulgurites is recognized to partly melt fragments of minerals and rocks that are incorporated into its structure [5]. The interior surface is smooth and glassy and may also contain trap gas inclusions from the surrounding area [6]. The chemical composition of fulgurites is depending upon the nature of the abundant minerals of surrounding environment [4] [5] [7]. The mineralogical composition (lechatelirite, cristobalite, chalcedony, opal) of fulgurites is related to the palaeo-environmental conditions of the semi-arid regions and to the melting conditions during the fulgurites forming lightning strike to the ground. Fulgurites are also very resistant to weathering and hence the study of properties of fulgurites may be helpful in understanding the ultra-high temperature natural phenomena during lightning [8]. Several putative impacts have been proposed based on the presence of shocked quartz in glassy samples alone [9]-[11]. Quartz is one of the most commonly used indicator minerals for shock metamorphism, as well as for pressure calibration. Quartz displays a wide variety of shock-induced mechanical deformations and transformations [12]-[16]. The studied fulgurites samples reveal the characteristic features of amorphous silica with some microcrystalline quartz and other minerals as trace [17] [18]. The structure of most SiO₂ polymorphous, both crystalline and amorphous, is based on SiO₄ tetrahedra. Amorphous silica is one of the polymorphous silica and at high temperature it can easily transform from quartz. In the Si-O-Si bending vibration region (400 - 700 cm⁻¹) of quartz, the band at 695 cm⁻¹ is determinative whether it is crystalline or amorphous [18]-[20]. In the present work, we address the infrared technique for estimation of micro-crystallinity of quartz in fulgurites. The sample has been characterized by powder X-ray diffraction, X-ray fluorescence and Fourier transform infrared (FT-IR) spectroscopic methods.

2. Materials and Methods

2.1. Fulgurite Samples

Fraction of two fulgurites samples are collected from Garuamukh near Nagaon town (latitude 26°20'N, longitude 92°41'E, Assam, India) and Kimin, near Itanagar (latitude 27°06'N, longitude 93°39'E, Arunachal Pradesh, India) for spectroscopic analysis. Each fulgurite sample comprising dark heterogeneous glassy material containing several white and greyish-brown areas (**Figure 1**). The total mass of the Garuamukh and Kimin fulgurite was estimated to be more than 10 kg and 30 kg respectively with numbers of individual pieces. On the basis of its physical attributes, these fulgurites can be classified as type II fulgurite.

2.2. Sampling and Analysis

A part of the samples are crushed into >90 μm, 90 - 63 μm, 63 - 45 μm and <45 μm fractions using mechanical sieve according to the Indian standard (IS 1607:2013) [21]. The smallest size achieved by a mechanical sieve is a 45 μm fraction. This fraction is sieved through a 45 μ sieve, where the <45 μm particles are obtained. We tried to minimize the grinding time to avoid the deformation of the crystal structure and the ion exchange. Powdered samples (0.25 g) are put into platinum crucibles and HNO₃ (conc.), HCl, H₂O₂ and HF are added in a proportion of 5:2:1:2 ml. Crucibles are heated on hot plate and the solution evaporated to near dryness. After that 2 ml HF are added in each samples few times until precipitate of SiO₂ is eliminated as SiF₄ vapors. After cooling down to the room temperature, a mixture of HCl (conc.) and redistilled water at a ratio 2:5 ml are added, the solutions transferred in 50 ml volumetric flasks and filled up with redistilled water. Then 0.5 g of powdered sample is put in a glass beaker and a mixture of redistilled water and HCl (conc.) in a ratio 15:20 ml is added and the solution evaporated to near dryness. The residue is dissolved with 10 ml 1% tin, and SiO₂ precipitated and coagulated. Precipitate is filtered and washed with HCl solution in a ratio 5:95 ml. Filter paper and residue are transferred into a platinum crucible and heated on 1000°C for 10 min. Crucible is weighed and the content of SiO₂ calculated. The different sized powdered sample was homogenized in spectrophotometric grade KBr (1:20) in an

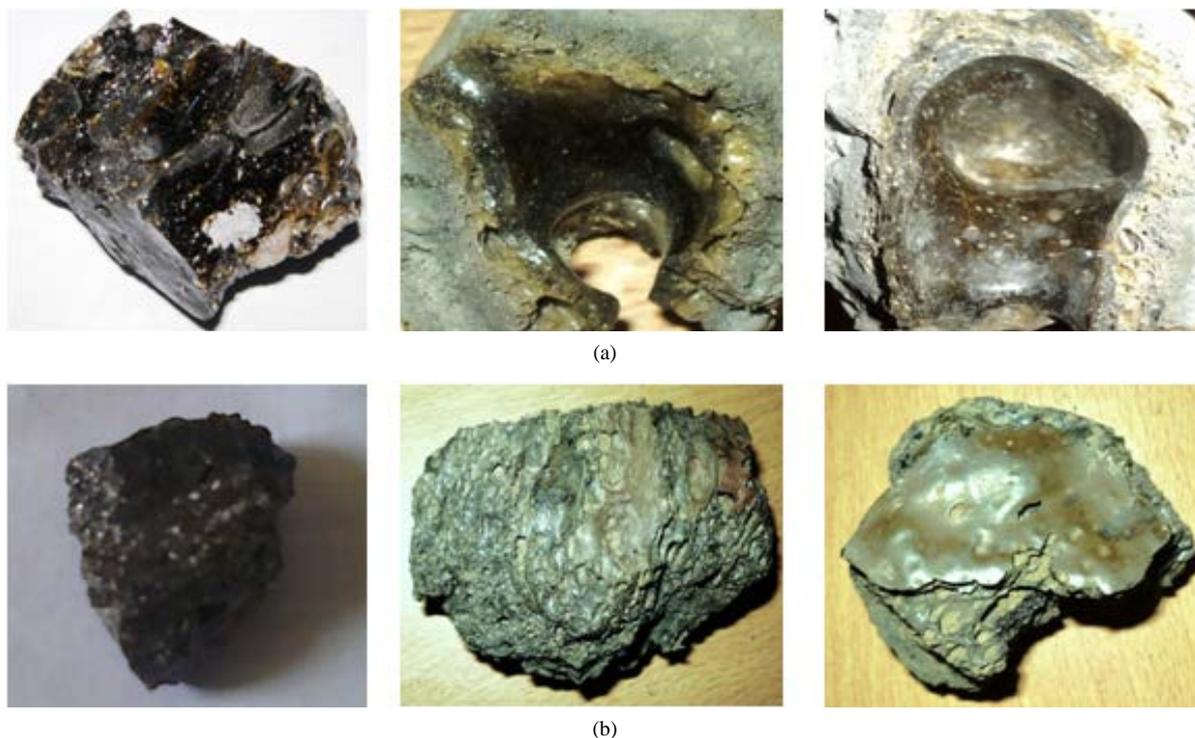


Figure 1. The photographs of three different fractions of Garuamukh and Kimin fulgurites. (a) Garuamukh; (b) Kimin.

agate mortar and was pressed with 3 mm pellets using hand press. The infrared spectra was acquired using Perkin-Elmer system 2000 FTIR spectrophotometer with helium-neon laser as the source reference, at a resolution of 4 cm^{-1} . The spectra were taken in transmission mode in the region $400 - 4000\text{ cm}^{-1}$. The room temperature was 30°C during the experiment. A part of samples (different particle sized) were mixed with a small amount of acetone and pipetted out onto a glass slide for X-ray diffraction (XRD) analysis. The glassy nature of the samples were confirmed by powder X-ray diffraction (XRD) analysis, by using the Philips PW 3710 XRD system which was operated at 40 KV and 45 mA between 5 and $75^\circ 2\theta$ at a scan speed of $0.04^\circ 2\theta/\text{s}$, with a $\text{CuK}\alpha$ ($\lambda = 1.54\text{ \AA}$) radiation and a graphite monochromator. The composition of the fulgurites was determined by using Philips X-ray fluorescence (XRF) machine. In X-ray fluorescence method, typical uncertainty involved in oxide analyses was about 0.01 wt%.

2.3. Crystallinity and Extinction Coefficient

The crystallinity indices are ascertained from the infrared spectra using the ratio of the optical densities of stretching mode at around 778 cm^{-1} and the bending mode at around 695 cm^{-1} . The optical density (D) is defined as the logarithm to the base 10 of the reciprocal of the transmitted radiant power (T). The estimation of the relative distribution of microcrystalline quartz in the studied samples is ascertained by calculating the extinction coefficient for the characteristic peak at 778 cm^{-1} using the relation:

$$\text{Extinction coefficient } (K) = DA/m$$

where A is the area of the pellet and m the mass of the sample [20].

3. Results and Discussions

3.1. Chemical Compositions of Fulgurites

Figure 1 presents the photograph of fulgurite samples, which is composed of dark heterogeneous glassy material containing several white and yellowish-brown spot regions with prominent gas bubbles trapped within glassy regions. The elemental compositions as determined by X-ray fluorescence spectroscopy of the Garuamukh and

Kimin fulgurites are presented in the **Table 1**. The compositional and mineralogical results show the major constituent of the fulgurites to be SiO₂ with miniature quantities of aluminium, titanium, iron, calcium and sodium oxides. The compositional results are compared with that of the previous work on fulgurites found elsewhere in similar occurrences [22]. The elemental composition of the samples (**Table 2**) exhibits low abundances of Ti, Na, Fe, Mg, K and Ca and relatively high abundances of Si and Al. The presence of CaO and Na₂O in the studied samples is indicative of plagioclase grain.

Table 1. Comparison of chemical compositions of the studied fulgurites with other fulgurites.

Occurrence Location	Composition (wt%)							
	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	TiO ₂	CaO	MgO	Na ₂ O	K ₂ O
Kimin (K-1) (<i>Present study</i>)	97.23	0.84	1.98	0.56	0.02	0.02	0.04	--
Kimin (K-2) (<i>Present study</i>)	97.10	0.75	1.98	0.40	0.02	0.01	0.02	--
Kimin (K-3) (<i>Present study</i>)	96.90	0.70	1.90	0.55	0.01	0.01	0.04	--
Kimin (K-4) (<i>Present study</i>)	97.31	0.69	1.88	0.47	0.02	0.01	0.02	--
Garuamukh (G-1) (<i>Present study</i>)	97.17	0.70	1.67	0.47	0.02	--	0.01	0.01
Garuamukh (G-2) (<i>Present study</i>)	97.00	0.61	1.85	0.40	0.05	--	0.05	0.03
Garuamukh (G-3) (<i>Present study</i>)	97.38	0.80	1.70	0.05	0.01	--	0.02	0.01
Garuamukh (G-3) (<i>Present study</i>)	97.40	0.80	1.73	0.05	0.01	--	0.02	0.01
Germany	93.80	--	3.80	--	0.60	--	--	--
South Amboy, New Jersey	99.00	0.30	0.70	--	--	--	--	--
Illinois	91.66	--	6.69	--	0.38	0.12	0.77	0.73
Holland	90.20	0.70	0.90	--	0.10	0.50	0.60	0.50
Libyan Desert	97.58	--	1.54	--	0.38	--	0.34	--
Little Ararat	58.59	4.47	21.29	--	6.36	2.49	4.42	0.94
Morocco	91.60	0.76	2.30	0.45	0.14	0.17	0.06	0.63
New South Wales, Australia	98.38	0.02	0.02	0.02	0.01	--	0.01	0.01
Quartzite, Arizona	59.70	5.50	16.4	0.87	6.40	2.30	2.70	3.70
Vernal, Utah	58.10	5.50	15.5	0.93	8.00	4.10	4.04	2.60
Chaffee County, Colorado	60.40	5.90	20.80	0.86	0.59	2.29	2.13	7.00

Results are compared with the findings of Frondel (1962).

Table 2. Elemental compositions (%wt) of Kimin and Garuamukh fulgurites.

Elements	Kimin					Gruamukh				
	K-1	K-2	K-3	K-4	Average	G-1	G-2	G-3	G-4	Average
Si	45.46	45.39	45.30	45.49	45.41	45.43	45.35	45.53	45.53	45.46
Fe	0.65	0.52	0.49	0.48	0.54	0.54	0.47	0.62	0.62	0.57
Al	1.05	1.05	1.01	0.99	1.02	0.88	0.98	0.90	0.92	0.92
Ti	0.34	0.24	0.33	0.28	0.30	0.28	0.24	0.03	0.03	0.15
Ca	0.01	0.01	0.01	0.01	0.01	0.01	0.04	0.01	0.01	0.02
Mg	0.01	0.01	0.01	0.01	0.01	--	--	--	--	--
Na	0.03	0.01	0.03	0.01	0.02	0.01	0.04	0.01	0.01	0.02
K	--	--	--	--	--	0.01	0.02	0.02	0.01	0.02

3.2. Characterization of Microcrystalline Quartz

Mixtures of identical amorphous and micro-crystalline phases has been identified or quantified in the fulgurites samples using X-ray diffraction. The X-ray diffraction patterns of the fulgurites samples are shown in **Figure 2** and different size fractions displays nearly constant diffraction pattern. Both quartz and cristobalite are often present in trace amounts in fused silica. The crystalline phases of micro-crystalline quartz and cristobalite are generally observed on the hump produced in the diffraction pattern by the amorphous silica. The broad hump

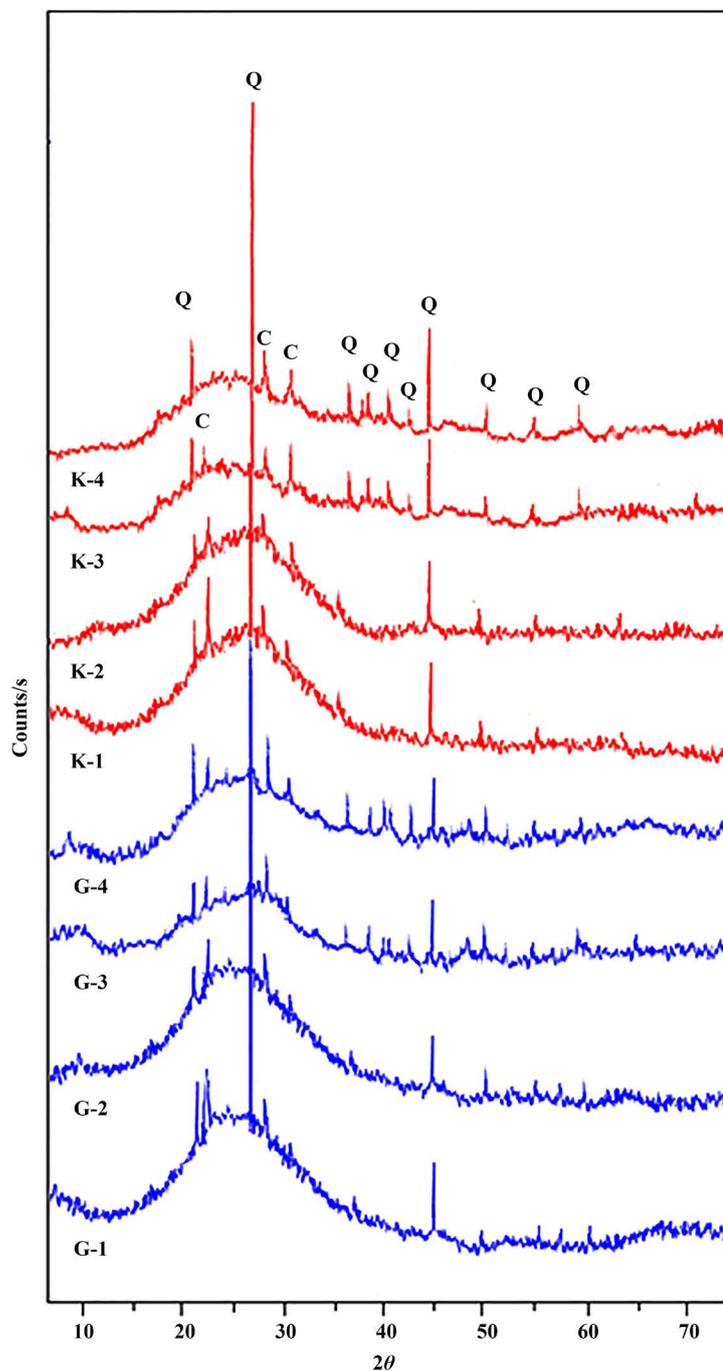


Figure 2. The X-ray diffraction patterns indicate quartz (Q) and cristobalite (C) phases in the Kimin and Garuamukh fulgurites.

observed in all spectra at around $18 - 36^{\circ}2\theta$ corresponds to a bulk composition of amorphous silica. Similar hump around $23^{\circ}2\theta$ in the Garuamukh fulgurites corresponding to the amorphous matrix of SiO_2 was also observed in the previous studies [18] [19]. The very strong peaks observed in all spectra at around $26^{\circ}2\theta$ are due to the microcrystalline quartz. Another strong feature of microcrystalline quartz is observed at $20.8^{\circ}2\theta$ and $45^{\circ}2\theta$. A strong feature at around $21.8^{\circ}2\theta$ in the spectra is reported as the presence of the silica polymorph cristobalite with further minor features between $28 - 31^{\circ}2\theta$ may be due to the presence of coesite. Further weak but distinct peaks of microcrystalline quartz were noted at around $36^{\circ}2\theta$, $39^{\circ}2\theta$, $40^{\circ}2\theta$, $42^{\circ}2\theta$, $50^{\circ}2\theta$, $54^{\circ}2\theta$ and $57^{\circ}2\theta$. The possible effects of the hematite inclusions corresponding to the peaks at around $36^{\circ}2\theta$ and $54^{\circ}2\theta$ cannot be ignored.

Figure 3 shows the infrared spectra of different particle sizes of the Garuamukh and Kimin fulgurites in the frequency range $500 - 2000 \text{ cm}^{-1}$. The mid infrared spectra of silicate in between the range $1200 - 400 \text{ cm}^{-1}$ are generally classified into four characteristic bands *i.e.* at $1080 - 1175 \text{ cm}^{-1}$ are arises due to Si-O asymmetrical stretching vibration (ν_3), $780 - 800 \text{ cm}^{-1}$ is arises due to Si-O symmetrical stretching vibration (ν_1), 695 cm^{-1} is arise due to Si-O symmetrical bending vibration (ν_2), and 464 cm^{-1} is arise due to Si-O asymmetrical bending vibration (ν_4) [23]. In the spectra of different particle sizes of fulgurites, the observed Si-O stretching vibration region at $785 - 787$, $952 - 965$, $1069 - 1080$, $1170 - 1174 \text{ cm}^{-1}$ are identical to the bands at 800 , 958 , 1088 cm^{-1} arise due to amorphous silica [24].

Among these four characteristic peak regions, the Si-O symmetrical bending vibrational peak at 695 cm^{-1} of the octahedral site symmetry is unique to the crystalline materials. It is well known that in the infrared spectra of amorphous silica the symmetrical bending vibration of the Si-O group found at 695 cm^{-1} is absent. Amorphous silica is one of the polymorphous of silica and at high temperature it can easily transform from quartz. Amorphous silica exhibits a relatively moderately strong peak at 800 cm^{-1} and it can be distinguished from the band of crystalline silicate [24]. Therefore, the symmetrical bending vibration of Si-O group obtained at 695 cm^{-1} is the diagnostic peak in determining the short range parameter of the quartz, whether it is crystalline or amorphous [20] [25].

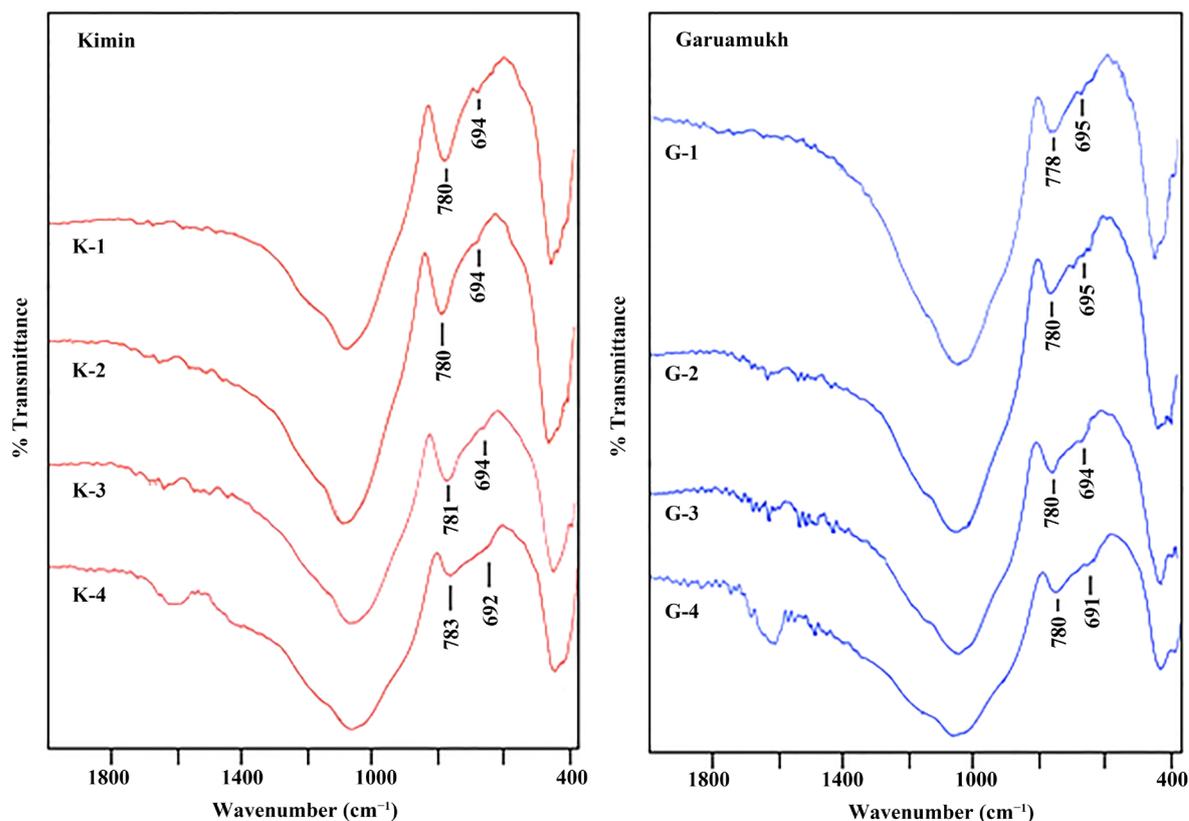


Figure 3. FTIR spectra of the Kimin and Garuamukh fulgurites in the range $2000 - 400 \text{ cm}^{-1}$.

A very weak smash around 695 cm^{-1} peak in the spectra of the both fulgurites of particle size $>90\text{ }\mu\text{m}$ is observed and this peak become gradually prominent as the particle size decreases to the finest range ($<45\text{ }\mu\text{m}$). These variations near 695 cm^{-1} peak in the spectra is indicative to the presence of micro-crystalline quartz contaminants which is also identical to the X-ray diffraction.

3.3. Distribution of Microcrystalline Quartz

The observed increases in infrared band strengths with decreasing particle size could be due to changes of crystallinity or optical behaviour. The tetrahedral Si-O symmetrical stretching vibrational peak at $780 - 800\text{ cm}^{-1}$ is stronger to that of the octahedral Si-O symmetrical bending vibrational peak at 695 cm^{-1} . Therefore, for any structural change, the damage occurs first in octahedral site symmetry then in tetrahedral site symmetry [24]-[26]. The intensity of the bands due to the vibrations of these two symmetries will provide direct information on the crystallinity. The calculated values of crystallinity index and extinction coefficient of different fraction sizes of the studied fulgurites are tabulated in Table 3. The particle size generally affects the characteristics peak of quartz [27]. At high temperature and pressure, silica polymorphs pass through a slow reconstructive recrystallization, during which existing Si-O bonds are broken and new bonds are made to allow atoms to move to appropriate positions in the more stable polymorph [28]. In glass ceramics, ceramics are produced by the controlled crystallization [29]. Any significant change in the crystallinity can be attributed to the geological conditions of formations and mechanical treatment such as grinding [30]. The optical densities of the samples are reciprocal to the calculated extinction coefficient (Table 3). Figure 4 displays the crystallinity index and extinction coefficients varies with the particle size. In the observed infrared spectra, the crystallinity is increases as the fraction size decreases but the X-ray diffraction does not reflect such pattern.

Table 3. Calculated value of optical densities of 778 cm^{-1} and 695 cm^{-1} , crystallinity indices and extinction coefficients of the different particle sizes of the fulgurites from infrared spectra.

Sample	Particle size (μm)	Optical density of 778 cm^{-1} peak	Optical density of 695 cm^{-1} peak	Crystallinity index	Extinction coefficient (K)
G-1	<45	0.571	0.544	1.049	89.69
G-2	45 - 63	0.566	0.543	1.041	88.85
G-3	63 - 90	0.560	0.540	1.036	87.98
G-4	>90	0.557	0.543	1.026	87.53
K-1	<45	0.628	0.550	1.142	98.69
K-2	45 - 63	0.601	0.537	1.119	94.41
K-3	63 - 90	0.590	0.553	1.066	92.68
K-4	>90	0.582	0.561	1.038	91.46

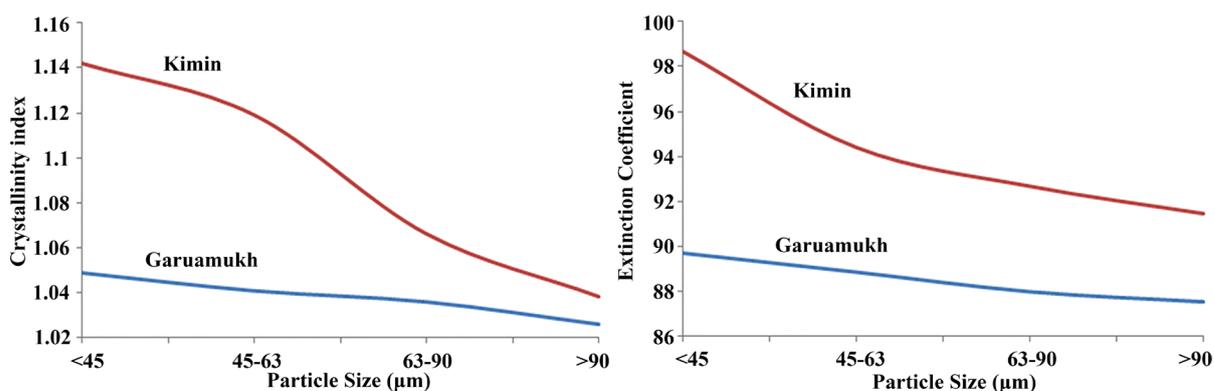


Figure 4. Variation of crystallinity index and extinction coefficient against the particle size in Kimin and Garuamukh fulgurites.

4. Conclusions

The compositional and mineralogical data as determined by X-ray fluorescence spectroscopy exhibit low abundances of Ti, Na, Fe and Ca oxides, and relatively high abundances of Si and Al oxides. These abundances are consistent with the composition of the target material of the fulgurites. The X-ray diffraction pattern containing the principal peaks for microcrystalline quartz and cristobalite and the overall pattern indicates the characteristic features of amorphous silica. Due to different melting and volatilization temperatures of quartz, Al-silicates and other ground mass minerals, an enrichment of other trace minerals may observe within the glassy matrix of the fulgurites.

The infrared spectra of glassy silicate materials of fulgurites are characterized by broad features that reflect the connectivity of the SiO₄ polymeric units within the three-dimensional framework of the silicate structure. Gucsik *et al.* also indicate the presence of the antisymmetric stretching vibration of the SiO₄ tetrahedra in 1043 - 1106 cm⁻¹ region of infrared spectra of natural glasses [31]. The presence of characteristic band at approximately 695 cm⁻¹ (ν_2 mode) in the infrared spectra is indicative of quartz. The frequency shifts in the Si-O symmetrical stretching vibration (ν_1) region of the studied infrared spectra are indicative to the presence of microcrystalline quartz. The ratio of the broad bending and stretching bands at approximately 695 cm⁻¹ and 778 cm⁻¹ can be used to elucidate glass nanostructure, composition, as well as temperature of formation. The structural changes take place in the octahedral symmetry in the formation of the sample, so the possibility of producing this silicate glass is by ultra high-temperature that is higher than the corresponding fusion point of the crystalline state, indicating the nature of fulgurites.

When the temperature generally quenched so rapidly to 600°C - 700°C, the SiO₄ tetrahedron could not be ordered to crystalline state and was preserved in an amorphous silicate phase. The presences of microcrystals in the occurrence of natural fulgurites have significant implications in the thermodynamic properties of shock metamorphism.

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