

## Research Article

# Characterization of Natural Precious Opal Using Modern Spectroscopic Techniques in Ethiopia: The Case from Delanta, South Wollo

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Received 31 May 2022; Revised 13 July 2022; Accepted 29 July 2022; Published 24 August 2022

Academic Editor: Lulu Zhao

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The purpose of this study was to conduct characterization of the Delanta natural opals obtained from different mining sites. Characteristics of opals mined from different geological locality were examined. Six opal samples were collected from the main mining sites found in different localities from Delanta district and characterized using Fourier infrared spectroscopy, scanning electron microscopy, X-ray diffraction, and inductively coupled plasma optical emission spectroscopy characterization techniques. The scanning electron microscope micrographs showed that all the samples have relatively semi-ordered (semi-crystalline) structural surface morphologies composed of tiny spherical particles. The performed X-ray diffraction analysis showed that the samples have main peaks at  $20.0^\circ$ ,  $21.72^\circ$ ,  $36.9^\circ$ ,  $44.0^\circ$ , and  $56.85^\circ$  with  $2\theta$  values between  $10^\circ$  and  $60^\circ$ . The five diffraction peaks are characteristic peak positions of opal-CT. The Fourier infrared spectroscopy data indicated that the prominent absorption bands for cristobalite at  $620$  and  $520\text{ cm}^{-1}$  were absent from the spectrum. Instead, three bands in the low frequency region centered at  $470$ ,  $791$ , and  $1100\text{ cm}^{-1}$  were observed because of the absorptions of the silicate framework (Si-O) vibrations. Moreover, spectra feature of molecular water and silanol (Si-OH) groups with broad absorption band centered around  $3447\text{ cm}^{-1}$  and the O-H stretching vibration of water molecules and water bending vibration with absorption band centered around  $1634\text{ cm}^{-1}$  were observed. These values were consistently comparable with literature data. The inductively coupled plasma optical emission spectroscopy analysis showed that four main impurities ( $>500$  parts per million) were Ca ( $\sim 1,750$ – $4,730$  parts per million), Al ( $\sim 1,990$ – $4,319$  parts per million), K ( $\sim 1,670$ – $3,895$  parts per million), and Na ( $\sim 595$ – $3,723$  parts per million). In general, this study revealed that all opal samples taken from six mining sites in Delanta district were found to be opal-CT as per the results of X-ray diffraction and Fourier infrared spectroscopy investigations.

## 1. Introduction

Opal gemstones are water-bearing non-crystalline materials, composed of microspheres of hydrated silica ( $\text{SiO}_2 \cdot n\text{H}_2\text{O}$ ) with a high degree of structural disorder [1, 2]. Because of their optical properties, opals have been widely used for jewelry, for fabrication of highly efficient optoelectronic devices, and as ornamental elements in art works [3–5]. Due

to high economic values and preciousness of opal resource, deep investigation and understanding of the physico-chemical properties of the mineral is an essential issue in gemstone industry.

The typical optical phenomenon of opal, called “play of color,” is as a result of the interaction of light with their pseudo-structure composed of regularly spaced layers consisting of sub-micron sized (approximately  $250\text{ nm}$  in

diameter) silica spheres [6–8]. The presence or absence of the optical phenomenon (play of color) in opals used to classify them as: precious opals those exhibit flash of spectral colors and common opals those do not exhibit flash of spectral colors. Even if common opals do not display play of colors, some common opals are attractive due to their opaque to translucent body color. While some rare opals are colorless, most opals present a body color: white, black, gray, brown, yellow to orange (as in fire opal), red, pink, blue, green, or violet, depending on mineral inclusions and on the existence of particular trace elements [9]. A number of these common opals have also significant commercial values. Opals vary in the crystallinity degree and in crystal-structure arrangement. Based on the mineralogical composition and atomic arrangements [10–12], opals can be classified into three types (opal-C, opal-CT, and opal-A) based on the main component revealed by X-ray diffraction (XRD), Fourier infrared spectroscopy (FTIR), Raman spectroscopy, and other techniques. Opal-C is a well-ordered form of silicate comprising mainly of  $\alpha$ -cristobalite; opal-CT is semi-crystalline and made up of crystalline regions of  $\alpha$ -cristobalite and  $\alpha$ -tridymite; and opal-A is the most disordered type..

In the literature, there are handful reports on the study of Ethiopian opals. Even the existing few reports lacked detail characterization studies of the Ethiopian opals particularly the Delanta opal, mined from the different mining sites. On the other hand, there are many studies on the Australian “potch” opal, which have attracted more interest on customers in global market. On the contrary, it is possible to find extensive studies of common opals of geological significance. In most of these studies, the classical mineralogical classification and the Raman classification are used.

The aim of this work was to characterize the Delanta natural opal dug out from different mining sites employing XRD, FTIR, SEM, and inductively coupled plasma optical emission spectroscopy (ICP-OES). As it is palpable, the characterization of the physicochemical properties of opals from a particular geographic area is important for several reasons. For instance, in the gem market, opals or other precious stones coming from specific place can be very valuable than others.

## 2. Experimental

**2.1. Sample Details and Preparation.** In order to conduct the detail characterization of the Delanta natural opals, 6 representative samples were collected from six main mining sites. The physical properties and photograph images of the samples are presented in Table 1 and in Figure 1, respectively.

After collection and leveling of the samples with different colors as shown in Figure 1, all the samples were powdered using crucible in chemistry department of Addis Ababa University and prepared for different characterizations as shown in Figure 2.

**2.2. Testing.** The surface morphologies of the samples were investigated by field emission scanning electron microscopy,

FEI Inspect F50, with working accelerating voltage of 15 kV and beam current of 140 nA at Addis Ababa Science and Technology University. Because of the inorganic nature of opal, a carbon coating was made to mount fragments to provide conductivity to avoid charging effects of electrons on the surface of the samples.

Powder X-ray diffraction data were collected at Addis Ababa University with MiniFlex 600 X-ray diffractometer equipped with Cu  $K\alpha$  radiation ( $\lambda = 1.5406 \text{ \AA}$ , 15 mA, 40 mV). The experiments were conducted in step-scan mode:  $10\text{--}60^\circ 2\theta$  range and step size of  $0.02^\circ$ . FTIR spectra measurements were recorded using a Perkin-Elmer BX Spectrometer ( $400\text{--}4000 \text{ cm}^{-1}$  spectral range) in KBr to determine the functional group of the samples. The elemental analysis was investigated using ICP-OES of model ARCOS FHS12. The experiments were carried out employing a plasma power of 1400 W and pumping speed of 30 rpm and argon pressure of 6.75 bars. XRD and FTIR experiments were recorded at Addis Ababa University, and ICP-OES measurements were performed at Horticoop Ethiopia (Debre-Zeit).

## 3. Results and Discussion

**3.1. Scanning Electron Microscopy (SEM).** From the SEM images of all opal samples, we tried to investigate the surface morphological behaviors. Figure 3 shows low magnification SEM images showing tiny spherical particles in opals; however, after 15–20 minutes of exposure of the samples to the electron rays and vacuum of the SEM, the water is lost and devitrification takes place. Consequently, microtexture with copious precipitation of tiny microspheres (Figure 3) agglomerated and constituted semi-crystalline heterogeneous sized stacked polyhedral microstructures. All the images showed relatively rough surfaces having different sizes of agglomerates with layer disposition.

Figures 3(a), 3(c), 3(e), and 3(f) show similar morphologies with comparatively bigger agglomerates. Particularly, Figures 3(c) and 3(e) show images with more phase separation between the particles. On the other hand, Figures 3(b) and 3(d) show smaller agglomerates and uniform particle size. In general, the SEM images show that all the samples have relatively certain semi-ordered structural surface morphologies composed of tiny spherical particles.

**3.2. X-Ray Diffraction Analysis.** It is well known that XRD is a powerful tool for structural characterization of materials in general and particularly very important for opal classification since the structural and spectroscopic properties of opals strongly depend on the crystallinity degree. The performed XRD analysis showed that all the samples have five diffraction peaks at  $20^\circ$ ,  $21.72^\circ$ ,  $36.9^\circ$ ,  $44^\circ$ , and  $56.8^\circ$  with  $2\theta$  values between  $10^\circ$  and  $60^\circ$ . The five diffraction peaks are characteristic peaks for opal-CT minerals according to the Jones and Segnit [10] classification and other literature as shown in Figure 4.

3), From the XRD diffraction spectra presented in Figure 4; the most intense peak occurs at about  $21.7^\circ$  with a very

TABLE 1: Natural opal samples collected from different mining sites of Delanta district and description of their physical properties.

S/N	Sample location	Sample code	Specific gravity	Hardness	Color	Appearance
1	Workwasha	Ds-1	1.92	5.5	Play of color	Yellow tint dominant blue reddish
2	Alehuat	Ds-2	1.90	5.5	Play of color	Brownish dominant reddish green
3	Nechgedel	Ds-3	1.95	5.5	Play of color	Blue reddish
4	Abobetach	Ds-4	1.90	6.5	Play of color	Brownish dominant green
5	Chegen	Ds-5	1.94	5.5	Play of color	Yellowish green
6	Gelbate	Ds-6	1.9	6.5	Play of color	Yellow tint dominant blue reddish

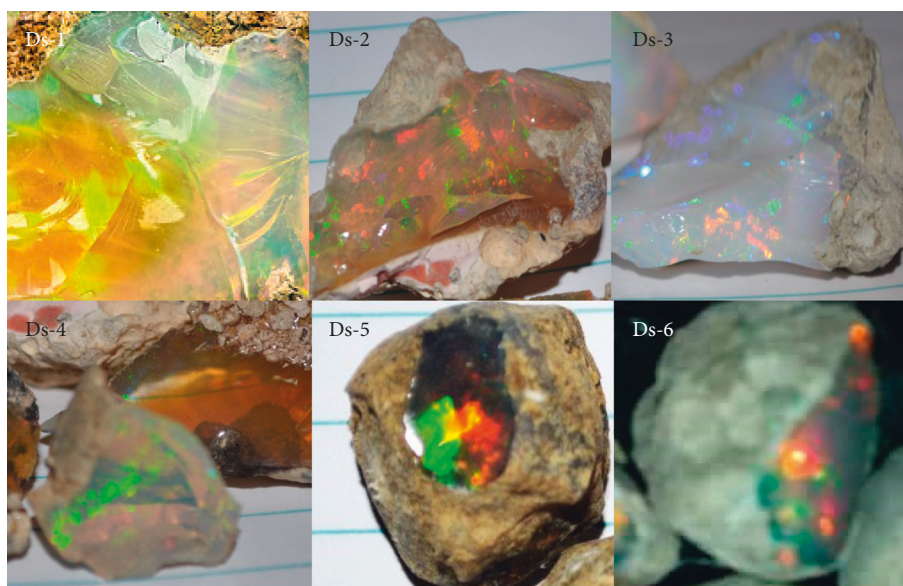


FIGURE 1: Photograph images of opal samples mined from different mining sites at Delanta district: (a) Ds-1, (b) Ds-2, (c) Ds-3, (d) Ds-4, (e) Ds-5, and (f) Ds-6.



FIGURE 2: Prepared powder natural opal samples for characterization.

weak shoulder at about  $20^\circ$ . There is also a weak reflection peak at about  $35.9^\circ$  resulting basically from  $\alpha$ -cristobalite with varying degrees of stacking disorder leading to the appearance of maxima which can be attributed to tridymite.

**3.3. FTIR Spectral Analysis.** Infrared absorption by mineral samples depends on the characteristics of the interatomic bonds in the mineral structure, the overall symmetry of the unit cell, and the local site symmetry of atoms within the unit

cell. The technique is responsive to both long-range and short-range order in minerals. IR spectra can, therefore, be used to characterize and identify both crystalline and amorphous materials.

Here, we report FTIR powder spectra, collected in the range of  $400\text{--}4000\text{ cm}^{-1}$ , in Figures 5 and 6. The results are totally well matched with those already available in the literature for related samples. Like most of the reported FTIR studies of opal-CT [10, 13, 14], this study showed that prominent absorption bands for cristobalite at 620 and 520



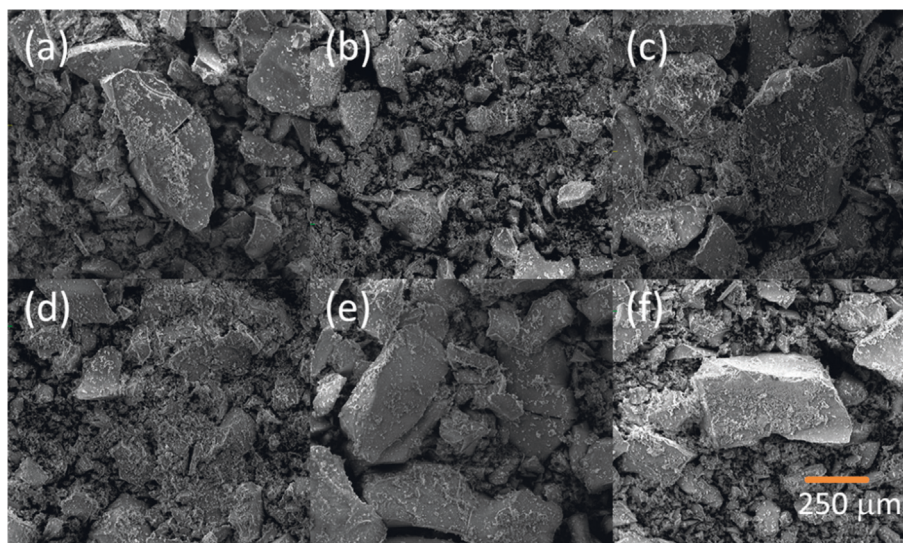


FIGURE 3: SEM images of opal samples: (a) Ds-1, (b) Ds-2, (c) Ds-3, (d) Ds-4, (e) Ds-5, and (f) Ds-6.

$\text{cm}^{-1}$  were absent from the spectrum [15] (Figure 5). All the samples taken from the different mining sites of Delanta district showed bands attributable to tridymite at 1100, 791, and  $470 \text{ cm}^{-1}$ . All these bands are related to vibrational modes within the silicon-oxygen framework [16] as indicated in Figure 5. The absence of absorbance at  $620 \text{ cm}^{-1}$ , which is generated by inter-tetrahedral Si-O vibrations (Figure 5), is predominantly critical information in determining the type of opals we are studying [17] as presented in Figure 6. Furthermore, as we can see from Figure 5, the bands at  $2921 \text{ cm}^{-1}$  and  $2852 \text{ cm}^{-1}$  are symmetrical and asymmetrical stretching vibrations of the methylene group C-H [18] bond in the material since our samples are natural opals. Likewise, all the samples exhibit absorption bands at approximately  $3447$  and  $1634 \text{ cm}^{-1}$  both attributed to O-H vibrations of water.

**3.4. Elemental Analysis.** The ICP-OES data for the six samples analyzed are listed in Table 2.

Trace element analysis is important for understanding some physical properties of gem opals, like color and luminescence [4]. The ICP-OES data of the six samples from six different mining sites of Delanta district are presented in Table 2.

As presented in Table 2, the four main impurities (>500 ppm) were Ca ( $\sim 1,750$ – $4,730$  ppm), Al ( $\sim 1,990$ – $4,319$  ppm), K ( $\sim 1,670$ – $3,895$  ppm), and Na ( $\sim 595$ – $3,723$  ppm). Other elements present in lesser amounts were (in order of decreasing concentration) Fe ( $\sim 70$ – $254$  ppm), Mn ( $\sim 3.0$ – $7.6$  ppm, except sample Ds-2 with  $1,157.8$  ppm), Zn ( $\sim 0.4$ – $2.0$  ppm), and Cu ( $\sim 0.04$ – $0.95$  ppm). The other impurity element tested from all the six samples was Mg. The result showed that three samples (Ds-1, Ds-3, and Ds-6) have Mg concentrations <  $0.002$  ppm. On the other hand, the other three samples (Ds-4, Ds-5, and Ds-2) have Mg concentration of  $\sim 47$ ,  $82$ , and  $172$  ppm, respectively.

The chemical composition of opals is commonly related to that of host rocks influences opal properties such as color and luminescence [4, 9]. The body color is typically associated with inclusion of colored minerals, whereas the abundance of some elements decides the color of the opal [4, 9]. Even if there are few reports [4, 19, 20] on the chemical composition and other spectroscopic analysis of the Delanta opal, the present study gives a detailed analysis focusing on specific mining sites in the district. When we see the concentration of elements in this study in comparison with a previous study reported by Gaillou et al., [4], even if the researchers did not mention the specific mining sites, the differences in concentration of some elements (Na, K, Fe) is significant. The concentration of Na samples: Ds-1, Ds-2, and Ds-3, Ds-4 is much greater than the concentration Na reported by Gaillou et al. [4]., [4]. When we compare the concentration of K, even if the K concentration of samples Ds-3 and Ds-5 is comparable with concentration values of the reported values, samples Ds-2 and Ds-4 of this study have much greater concentration values. But when we compare the concentrations of Ca, Mg, Mn, Cu, and Zn, the concentrations we get are comparable with the concentrations reported by Gaillou et al. [4] even if Mg was not detected from three samples of this study. The detection of Mn in all of the samples is probably due to the presence of dendrites of todorokite.

Sample Ds-2 from Alehuat is identified and differs from other samples by the highest concentration of Mn, Fe, Mg, and average value of Zn ( $1,157.83$ ,  $254.39$ ,  $172.38$ , and  $1.58$ ), respectively.

Generally, in this study, we observed that elements Zn, Mn, and Cu have low concentrations in all the six investigated samples and their presence does not look related to the color of the opals. When we observe the concentration of Fe, it is reasonable to associate with the color of the samples: the brownish dominated color of sample Ds-2 is due to the highest concentration of Fe, whereas the lower Fe concentrations in

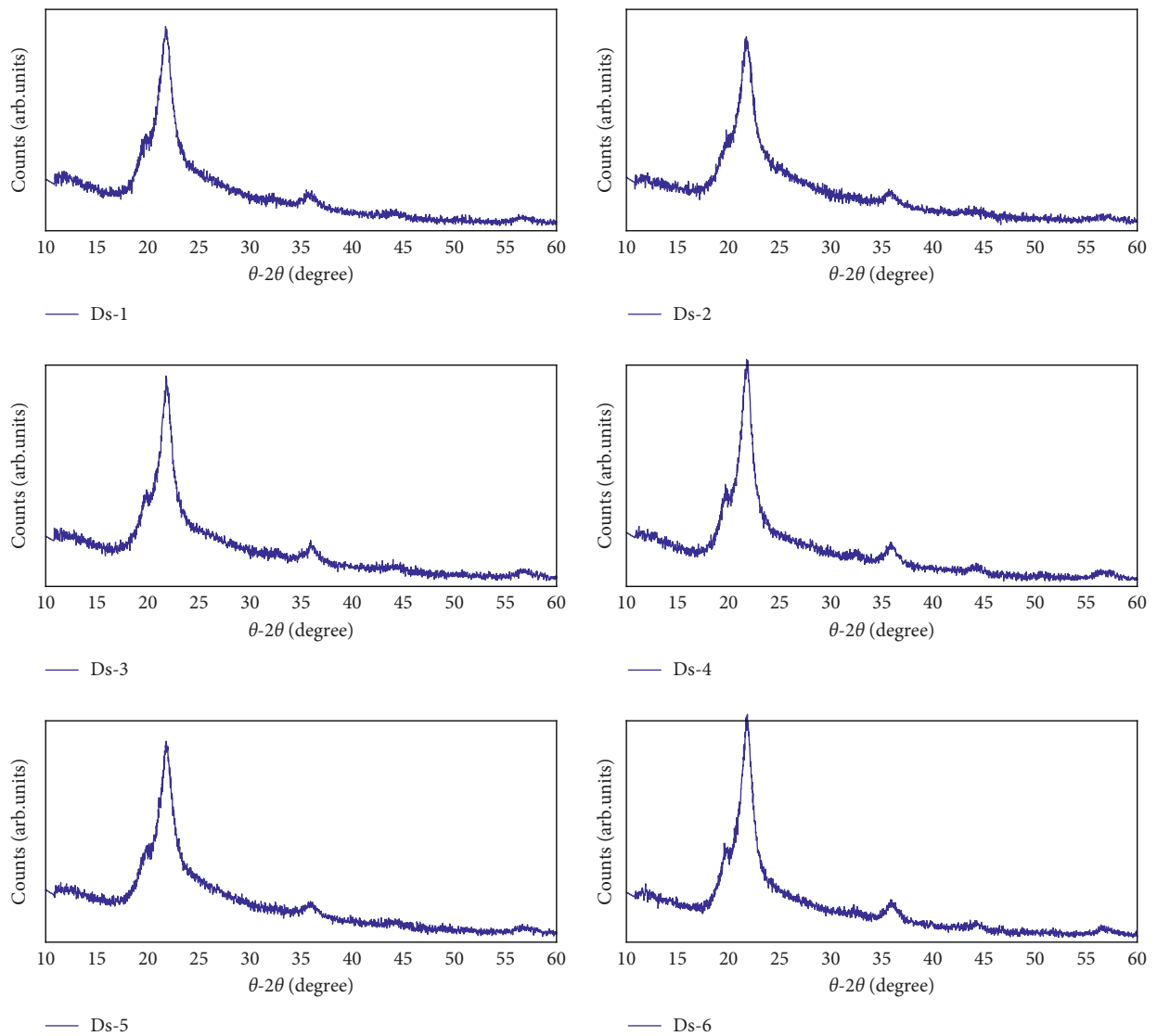


FIGURE 4: XRD diffraction patterns of opal samples mined from different geological localities of Delanta.

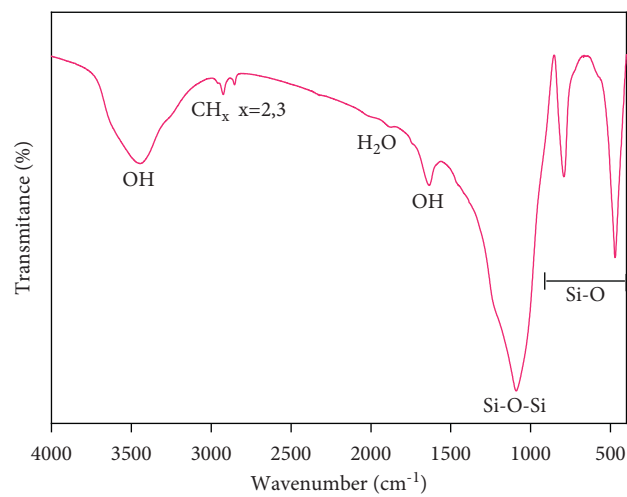


FIGURE 5: FTIR representative spectra profile of opal samples from Delanta district taken from mining site Workwasha.

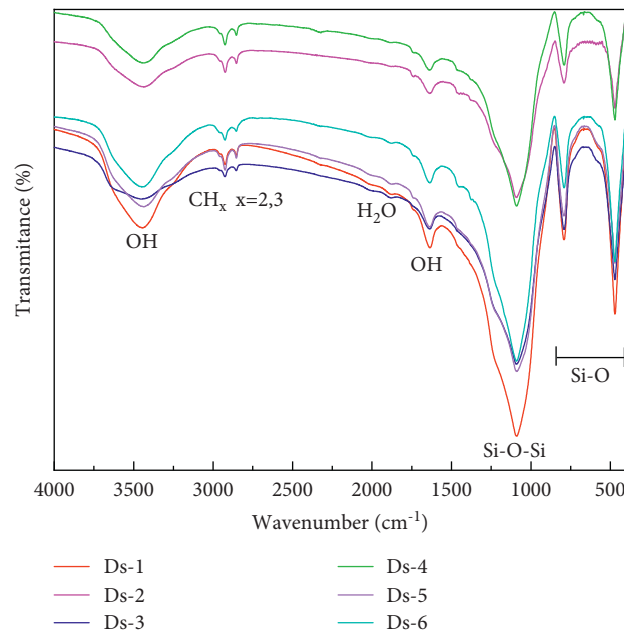


FIGURE 6: FTIR spectra profiles of opal samples: Ds-1, Ds-2, Ds-3, Ds-4, Ds-5, and Ds-6.

TABLE 2: Concentration (in ppm) for trace elements within each opal sample by ICP-OES (Ds-1: Workwasha, Ds-2: Alehuat, Ds-3: Nechedel, Ds-4: Abobetach, Ds-5: Chegen, and Ds-6: Gelbate).

	Ds-1	Ds-2	Ds-3	Ds-4	Ds-5	Ds-6
Ca	2,258.46	4,338.71	1,756.16	4,737.35	2,402.43	2,782.30
Al	2,634	3,152	4,287.51	2,934	4,319	1,990.78
Mg	<0.002	172.38	<0.002	47.04	81.92	<0.002
Na	3,723.24	1,130.13	1,995.66	2,019.39	595.18	3,376.62
K	2,466.55	3,416.98	1,670.00	3,894.75	1,840.77	2,461.04
P	478.91	364.58	415.69	438.16	353.58	251.51
Fe	70.31	254.39	185.95	226.41	183.93	86.50
Mn	4.91	1,157.83	7.11	7.65	2.99	4.90
Cu	0.95	0.65	0.41	0.32	0.04	0.19
Zn	1.82	1.58	1.88	1.87	1.29	0.44

opal samples Ds-1 and Ds-6 determine the yellow tint color. This relationship between Fe concentrations with the colors of opal samples was also reported by different authors including Gaillou et al. [4]. The existence of the impurity Fe in opals of the study area perhaps occurs as inclusion of Fe oxyhydroxides that determine the yellow or orange colors [4]. When we see the variations of elemental concentrations in the samples, the variation of Ca and K is highly significant relative to the variation of concentrations of other elements with the lithological origins of the samples and can then be valuable to determine the provenance.

Some optical and structural properties (color, preciousness, and atomic arrangement) of opal samples mined from different mining sites of Delata distinct are compared

with the same properties of opals from other countries (Australia, USA, Slovakia, Madagascar, and Mexico) from the literature [21] as presented in Table 3. Compared with the listed countries, opals taken from different locations of Delata district are all precious with different colors (Figure 1). But many opal samples taken from different locations of the aforementioned countries are common with almost one type of color. Some precious opals are seen in few locations of Australia, USA, and Slovakia. When we consider the classification of opal samples based on atomic arrangements, all the samples taken from Delata district are opal-CT. But in addition to opal-CT, opal-A is found in Australia and Slovakia, and opal-C is mainly found in Mexico.

TABLE 3: Comparison of some optical and structural properties of Delanta district opals with other countries' opals [21].

	Sample	Location	Color	Classification	Opal type
Ethiopia	Ds-1	Workwasha	Yellow dominant blue reddish	Precious	CT
	Ds-2	Alehvat	Brownish dominant reddish green	Precious	CT
	Ds-3	Nech-gedel	Blue reddish	Precious	CT
	Ds-4	Abobetach	Brownish dominant green	Precious	CT
	Ds-5	Chegen	Yellowish green	Precious	CT
	Ds-6	Gelbate	Yellow dominant blue reddish	Precious	CT
Australia	AU047	Andamooka	Yellow	Common/precious	A/CT
	AU008	Andamooka	White	Common	CT
	AU110	Andamooka	Uncolored	Common	A
	AUM8	Andamooka	Yellow	Common/precious	CT
	AU120	Cooper Pedy	Turquoise	Common	A
USA	U1240	Virgin Valley	Black	Precious	CT
	U01	Humboldt Valley	Yellow	Common	CT
Slovakia	SL01	Dubník	White	Precious	A
	SL03	Herlany	Opaque red	Common	CT
	SL1243	Košice	Uncolored	Common	CT
Madagascar	MA1211	Unknown	Green	Common	CT
Mexico	MX1403	Unknown	Milky yellow	Common	C
	MX1233	Hidalgo	Uncolored	Common	CT

#### 4. Conclusion

The main purpose of this study was to conduct characterization of natural opal samples (all precious and collected from different mining sites) from Delanta district, South Wollo, Ethiopia. The study provides a classification of samples based on the characterization results. So as to draw a clear picture of what has been done, the main conclusions are summarized as follows.

Six colored raw opals (all play of color) were collected from different lithological representative mining sites of Delanta district. To make the samples ready for characterization, we removed the soil from the body of all the samples and the samples were crushed using crucible. In this study, SEM, XRD, FTIR spectra, and ICP-OES methods were employed to study the morphological, structural, and chemical properties of the samples. The SEM micrographs showed that all the samples have relatively semi-ordered structural surface morphologies composed of tiny spherical particles. However, samples taken from Alehvat and Abobetach showed relatively smaller agglomerates and uniform particles sizes. As it is palpable, XRD is one of the appropriate analytical methods to classify opals as opal-A, opal-CT, and opal-C. The performed XRD analysis showed that all samples are polycrystalline and have five diffraction peaks at  $20^\circ$ ,  $21.72^\circ$ ,  $36.9^\circ$ ,  $44^\circ$ , and  $56.8^\circ$  which are characteristic XRD diffraction patterns of opal-CT indicating the play-of-color nature of samples since play-of-color opals belong to either the opal-A group or opal-CT group. Likewise, the FTIR spectra characterization showed five main peaks known for opal-CT in the literature ( $1100$ ,  $791$ ,  $470$ ,  $2923$ , and  $2852\text{ cm}^{-1}$ ). Generally, both XRD and Raman spectra measurements showed with good agreement that the samples collected from all sites are opal-CT. The ICP-OES analysis showed that four main impurities ( $>500\text{ ppm}$ ) were Ca ( $\sim 1,750\text{--}4,730\text{ ppm}$ ), Al ( $\sim 1,990\text{--}4,319\text{ ppm}$ ), K ( $\sim 1,670\text{--}3,895\text{ ppm}$ ), and Na ( $\sim 595\text{--}3,723\text{ ppm}$ ). Other elements

present in lesser amounts were (in order of decreasing concentration) Fe ( $\sim 70\text{--}254\text{ ppm}$ ), Mn ( $\sim 3.0\text{--}7.6\text{ ppm}$ , except sample Ds-2 with  $1,157.8\text{ ppm}$ ), Zn ( $\sim 0.4\text{--}2.0\text{ ppm}$ ), and Cu ( $\sim 0.04\text{--}0.95\text{ ppm}$ ).

#### Data Availability

The data used to support the findings of this study are included in this paper. If additional data is needed, the authors can provide.

#### Conflicts of Interest

The authors declare that they have no conflicts of interest.

#### Acknowledgments

This study was supported by Wollo University (wu-2012).

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