



# Spectrochemical Analysis of Lonar Lake Water and Sediment using dc-glow Discharge Technique

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## Abstract

*The dc-glow discharge technique was employed to study the excitation mechanism of water (brine) and sediment of the Lonar crater. The elemental analysis of the crater water and sediment was carried out. The yield of Fe from the crater water was obtained by the dc glow discharge. The spectra of the Lonar water and sediment sample emitted during glow discharge are recorded using PMT and Monochromator. The various emission peaks in the region 400 to 480nm of the spectrum indicate the contamination of the different metals Fe, Ni and Ti in sediment sample. The percentage of Fe 1.946 % and of Ni 6.842 % is estimated gravimetrically from the sediment sample in the Lonar crater.*

**Key Words:** Lonar crater; dc-glow discharge; spectrochemical

## Introduction

Among the biggest crater in the world, the third one is Lonar Crater in India, which is due to impact of meteorite in Basaltic rock. The Lonar crater (Latitude: 19° 59' N and Longitude: 76° 31' E), in the Buldana District of Maharashtra (India) is a Circular Lake occupied by saline water located in Deccan trap [1-3]. The lake water is alkaline, having pH about 10.5, which is ten times saltier than drinking water. Alkalinity of the lake is mainly due to the higher percentage of Sodium Carbonate and hence it was used previously as a source of washing soda [4].

Lonar crater, India, is one of the youngest and best preserved impact structures on earth. The geochemical analysis of metallic contamination of the sediment of Lonar crater has a great significance. The geochemical study of six target basalts and six melt rock samples show that latter were enriched in the abundance of Al, Fe, K, Co, and Sr, depleted in those of Ti, Mg, Cr and Sc, as compared to the basalt compositions [5]. Geochemical study of Lonar impact rocks reported the presence of small (<10 $\mu$ m) iron-nickel particles with wide range of Fe/Ni ratio (0.02-14.4) within the vesicles of Lonar impact melt [6]. Microprobe analysis of melt rich and magnetite show these spherules are distinct from average target basalt and relatively high Fe<sub>2</sub>O<sub>3</sub> and MgO and low Na<sub>2</sub>O, K<sub>2</sub>O and distinctly higher Cr, Co and Ni content. The Ni content shows the largest variation from ~ 60 to 2500 ppm in these spherules [7].

The microprobe analysis reported that the sub-millimeter size spherules contains relatively higher percentage of Cr and Ni and concluded that spherules were probably formed by mixing of target basalt melts and chondrite impactor materials [8]. The mossbauer spectroscopic study of iron mineralogy of eight impactite samples of the Lonar crater shows significant amount of Fe<sup>3+</sup> phase and besides Fe<sup>3+</sup> the sample exhibited a strong Fe<sup>2+</sup> doublet corresponding to main iron containing mineral [9]. The geochemistry of Deccan basalt has been shown to be similar to martian meteorite basalts containing higher quantities of Fe and lower Al than most terrestrial basalt [10].

## Experimental

The sample of crater water (brine) of pH 10.27 is collected from the crater. The samples of sediment are collected from different regions of the crater. The experimental technique for carrying out voltage-current characteristics of collected lake samples is presented in figure 1.

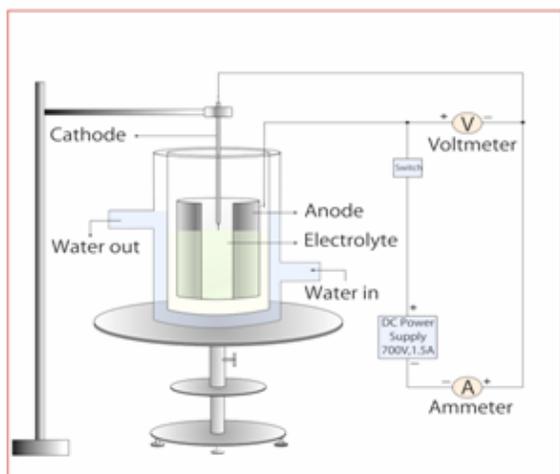


Fig. 1. Dc-glow discharge technique.

The tungsten electrode of diameter 3 mm suspended in a hollow stainless steel cylinder of length 6 cm, and internal diameter 2.54 cm. The whole arrangement is placed in a glass beaker having capacity 100 ml. Using sample as the anode and cathode, the voltage-current characteristics and total intensity emitted during glow discharge is recorded. The quantitative chemical analysis of the brine and sediment sample is carried out.

## Result and Discussion

The electrolytic process leading to the luminescent glow at the interface of tungsten electrode, the samples of brine and sediment solutions are used as an anode and a cathode.

### *Lake Water as an Anode*

The discharge current and intensity of the glow discharge corresponding to applied voltage across the solid liquid interface for brine as an anode and cathode is displayed in table 1.

Table 1. Discharge currents and Intensities for corresponding applied voltage across electrodes of glow discharge for Crater water.

Brine as an Anode			Brine as Cathode		
Voltage (V)	Discharge current (A)	Intensity (A.U.)	Voltage (V)	Discharge current (A)	Intensity (A.U.)
25	0.15	-	28	0.025	-
30	0.175	-	48	0.05	-
35	0.2	-	60	0.075	-
40	0.225	-	72	0.1	-
45	0.25	-	130	0.075	-
50	0.275	-	170	0.05	-
55	0.3	-	190	0.075	-
60	0.275	-	215	0.1	0.02
70	0.25	-	250	0.125	0.04
75	0.175	-	290	0.15	0.06
80	0.125	-	300	0.175	0.1
100	0.1	0.02	-	-	-
125	0.125	0.1	-	-	-
150	0.175	0.17	-	-	-
175	0.25	0.3	-	-	-

Figure 2 displays the standard voltage-current characteristics and variation of intensity of glow for brine is used as an anode.

The liner section AB represent the applied voltage across the cell is directly proportional to the discharge current which satisfies the Ohms law. In the region B to C voltmeter and ammeter widely fluctuates and electrolytic process is unstable. The rate of migration of ions decreased and charge transformation at the interface between the solid and liquid electrodes is obstructed with fall in current. At point C the fluctuation suddenly stopped leads to the local heating at the interface. On further increase in voltage, produces intermittent sparking of orange color. The local heating process then forces out a vapor jet and nearby liquid molecules speedy

there. The region CD of the curve represented the situation. The region BC indicate negative resistance region. At point D slope of the curve changes the sign and the formation of stable superheated insulating layer around the electrode causes continuous yellow glow at the voltage greater than 100V. The intensity of glow increases monotonously with the current.

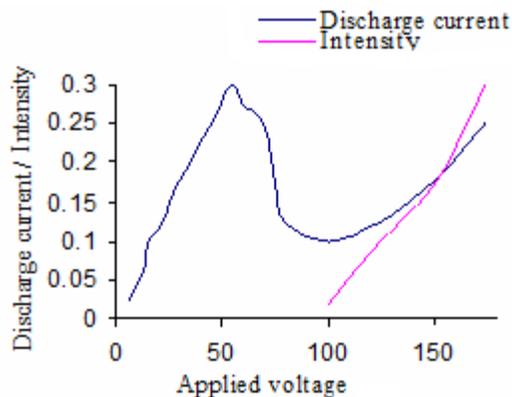


Fig. 2. V-I characteristics of crater water as an anode.

### Lake Water as a cathode

The nature of V-I curve is similar for brine as a cathode. Figure 3 depicts the same. The glow discharge appears at significantly high voltage greater than 170 V as compared to it used as an anode. The orange and yellow color plasma in the glow discharge indicates the spectral signature of alkali metals and alkaline earth metals like Na and Ca. further increase of voltage, plasma color changed to faint sky and it appear as violet, indicates the abundance of transition metals.

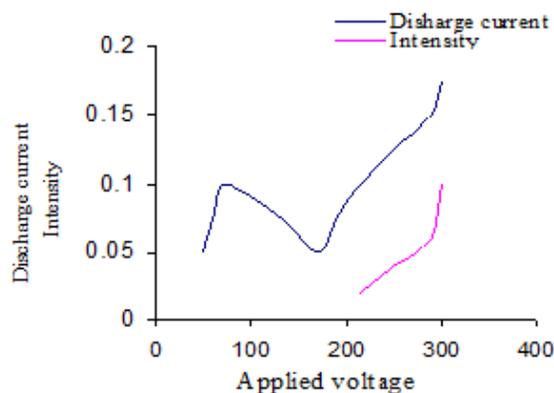


Fig. 3. V-I characteristics of crater water as a cathode.

### Sediment Sample used as anode and cathode

Dried powder of sediment sample (10gm) dissolved in distilled water +HCl in (1:1) ratio and 100 ml solution is prepared. The discharge current and intensity of the glow corresponding to applied voltage across the solid liquid interface for brine as an anode and cathode is displayed in table 2. The V-I characteristics of the sediment solution using as an anode and cathode is determined. The figures 4 and 5 depict the same. The dc-glow discharge appeared at voltage greater than 35V and orange intermittent sparking with decreasing current appeared. Above 35V the faint sky plasma appeared with decreasing current. At 50V the color of plasma changes from faint sky to lavender. Above 50V the current changes its sign i.e. it goes on increasing and plasma color changed to violet. Further increasing the voltage the intensity of violet plasma increased with current monotonously. The violet plasma appeared for sediment sample used as a cathode. Different plasma colors confirm the abundance of various transition metals like Fe, Ni and Ti in the sediment of crater.

Table 2: Discharge currents and Intensities for corresponding applied voltage across

electrodes of glow discharge for Sediment sample.

Sediment as an Anode			Sediment as Cathode		
Voltage (V)	Discharge current (A)	Intensity (A.U.)	Voltage (V)	Discharge current (A)	Intensity (A.U.)
4	0.125	-	7	0.025	-
6	0.2	-	10	0.05	-
8	0.25	-	12	0.075	-
10	0.325	-	40	0.15	-
15	0.5	-	45	0.1	-
20	0.55	-	60	0.05	0.02
25	0.5	-	100	0.075	0.07
30	0.45	-	200	0.1	0.08
35	0.4	-	290	0.25	0.12
40	0.425	0.02	-	-	-
50	0.5	0.07	-	-	-
75	0.6	0.1	-	-	-
85	0.675	0.15	-	-	-
100	0.725	0.22	-	-	-
110	0.8	0.36	-	-	-

### Yield of Iron from brine by dc-glow discharge

In the same experiment brine is used as an anode. The dc-voltage is applied in equal steps. The conventional electrolysis occurred with small bubbles of gas leaving the cathode. During the electrolysis the reddish brown precipitate is formed. The precipitate is tested by semi-microanalysis and Fe(OH)<sub>3</sub> is confirmed. The precipitate is ignited in silica crucible for 5-6 hours and weighed as Fe<sub>2</sub>O<sub>3</sub>. The crucible is allowed to cool in a desiccator. From Fe<sub>2</sub>O<sub>3</sub> weight of Fe is calculated.

### Geo-chemical behavior of sediment sample

The semi microanalysis of the sample is carried and confirmed the presence of Fe, Ni and Ti. Iron and Nickel is estimated gravimetrically. The solid material of iron oxide and nickel dimethyle-glyoxime complex weighed in a silica crucible as per routine gravimetric analysis and found 1.946 % pure iron and 6.842 % of nickel.

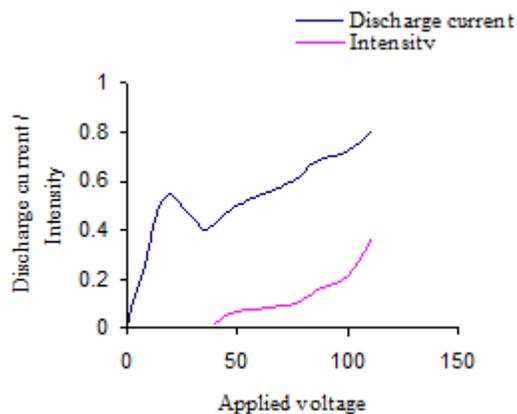


Fig. 4. V-I characteristics of sediment of crater as an anode.

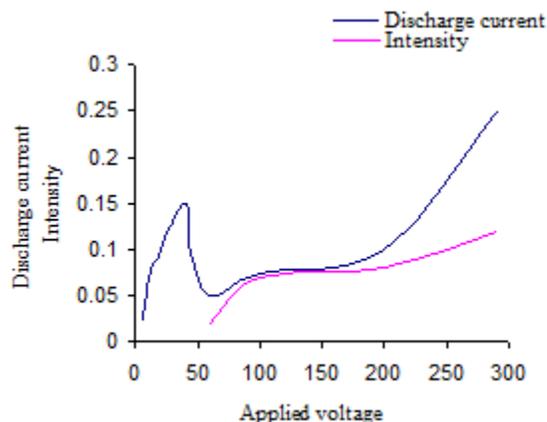


Fig. 5. V-I characteristics of sediment of crater as a cathode.

### GSpectroscopic variation of intensity as function of wavelength in the glow discharge

The variation of intensity with wavelength was studied for determination of the chemical contents of crater samples. We employed photomultiplier tube (PMT) interfaced with monochromator as a spectrum analyzer. Figure 6 displays the emission spectra of variation of intensity v/s wavelength of discharge plasma. The various peaks in the spectrum of the water and sediment attributed to alkali, alkaline earth metals and different transition metals.

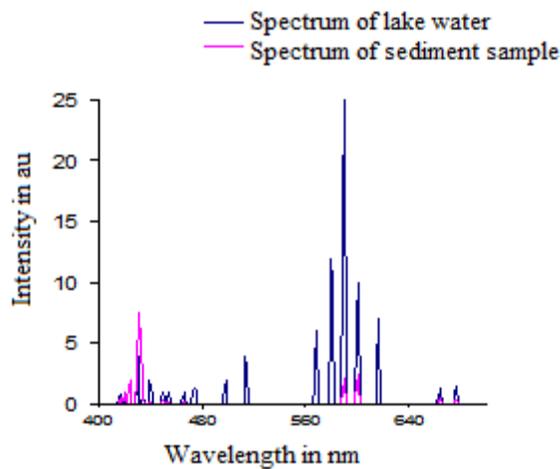


Fig. 6. Emission spectra of lake water and sediment sample.

The sample of brine exhibited prominent higher intensity emission lines in the region 580 to 620 nm these bands attributed to alkali and alkali earth metals Na and Ca respectively. Sodium and calcium emission lines are specified from Atomic Energy Levels Vol. I [13]. The similar lines of emission are depicted in the analysis of drinking water [14]. The abundance of these metals concluded that the lake water is highly saltier and alkaline. The salinity and alkalinity of lake water is attributed to higher concentration of salts of Na and Ca like  $\text{Na}_2\text{O}$ ,  $\text{Na}_2\text{CO}_3$ , and  $\text{CaCO}_3$  respectively. The similar finding are depicted in the work of N. C. Nandy et al. and Jhingran A.G. et al [1,4]. The sediment sample exhibited two significant lower intensity peaks at 588 and 590 nm as compared to lake water, corresponding to very rare sodium containing minerals.

The brine sample exhibited very few lower intensity emissions in the region 400 to 500 nm compared to the sediment sample. The higher intensity peaks exhibited by sediment sample in the region 400 to 470 nm corresponding to main transition metals like Ni, Cr, Fe and Ti containing minerals. The four bands at 425, 430, 440 and 466 nm are

attributed to transitional metal Fe, The Fe emission lines at 425.1, and 430 nm are significantly noted in the determination of manganese and chromium in steel with a dual cathode glow discharge lamp [15]. The four significant lines lead to the conclusion that the sediment containing Fe-rich minerals. Similar findings are seen in the work of Hagerty J. J. et al [10]. The emission lines at 416 and 420 nm are assigned to element Ni. The wavelengths of emission of Fe and Ni are specified from Atomic Energy Levels volume II [16]. The sample exhibited Fe and Ni emission lines, however most interesting characteristic of the study is higher intensity emission at 432 nm corresponding to main Titanium containing mineral in particular sediment sample of Lonar Lake. The Ti emission line is predicted from Atomic Energy Levels Vol. I [13]. The presence of titaniferous magnetic crystals is predicted in the study of samples of Lonar Impact melts [17].

## Conclusion

In the view of the result discussed, the comparative study of brine and sediment sample of Lonar crater it is concluded that dc-glow discharge method is superlative and very economical technique for confirmation of metallic contamination of the samples. The excitation of different plasmas in the discharge obtained at atmospheric pressure in air indicates the abundance of Fe, Ni and Ti in the sediment sample of the Lonar crater.

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