

Composition and morphology study of acid-digested pond ash

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The present study examines the chemical composition and morphology of raw and acid-digested pond ash samples that were collected from four ash ponds of the Fertilizer Corporation of India Limited, Sindri unit, Jharkhand, India. Scanning electron microscopy and energy dispersive X-ray spectroscopy were used to analyse the four pond ash samples. The aim of the study was to characterize the pond ash samples in order to assess their utilization based on morphological characteristics. Besides, loss-on-ignition and leaching studies (both with distilled water and under acidic medium conditions) were also conducted to understand the mobility of various elements in the leachates. The information provided herein would be useful to clearly understand the difference in the chemical composition and morphology of the raw and acid digested pond ash samples.

Keywords: Chemical composition, elemental analysis, morphology, pond ash.

INDIA, on account of its huge amount of coal reserves, is dependent on coal for the generation of electricity. More than 50% of India's electricity comes from the combustion of coal at thermal power stations^{1,2}. This process produces inorganic residues commonly known as coal combustion residues (CCRs). Over 100 million tonnes of CCRs (fly ash – 80% and bottom ash – 20%) are generated annually from the existing more than 100 thermal power stations³⁻⁷. The physical and chemical characteristics of CCRs are controlled by the coal type, boiler and its operating conditions, and post-combustion parameters⁸. The ash utilization in 1992–93 was 2–3% (ref. 6). The utilization statistics of CCRs has been estimated as more than 50% (refs 5, 9). This is not enough if one compares it with the developed countries of the world, where utilization percentage is more than 70 and in few cases even close to 100 (refs 6, 9).

During combustion, minerals in coal become fluid at high temperature and are then cooled. In a pulverized coal (PC)-fired boiler, the furnace operating temperatures are typically in excess of 1400°C. At these temperatures, the mineral matter within the coal may oxidize, decompose, fuse, disintegrate or agglomerate¹⁰. Rapid cooling in the post-combustion zone results in the formation of

spherical, amorphous (non-crystalline) particles. Expansion of trapped volatile matter can cause the particle to expand to form a hollow cenosphere¹⁰⁻¹². Minerals with high melting point may remain relatively unchanged. The heating and cooling have a significant effect on the composition and morphology of each particle^{10,13,14}. Some researchers conclude that fly ash cenospheres are good filler material¹⁵. They can be used for improving the properties of nylon-6. They can also be used to produce a new class of engineering composites for automobile applications. Being light-weight and free-flowing in nature, fly ash cenospheres have varied applications in ceramics, plastics, paints and construction.

For ash generated from thermal power stations, the original composition of the feed coal, combustion conditions, size of the fly ash particles, and fly ash mineralogy influence the distribution and mobility of trace metals. Leaching of trace elements in the environment from disposed ash is a matter of great environmental concern and requires attention. Analytical approach is used for characterization of such ash in order to understand the chemistry and mineralogy. Leaching studies also need to be carried out to know the mobility of various trace and heavy elements into the water environment resulting in likely contamination of surface water and groundwater. Several studies have been carried out on the leachability of elements. Some¹⁶ researchers have studied the leachability of heavy elements like Cu, Pb and Zn while others have studied the leachability of Ag, As, Ba, Ca, Cd, Co, Cr, Cu, Fe, Mg, Mn, Mo, Ni, Pb, Sb, Se, Sr, Ti and Zn¹⁷ from fly ash samples taken from three different low-grade lignite-fired stations.

The aim of the present study was to use scanning electron microscopy (SEM) and energy dispersive X-ray spectrometry (EDS) to characterize the raw and acid-digested pond ash samples according to their morphology and composition. SEM images help us understand the distinct spherical nature of the grains for the fly ash samples, while composition indicates the type of ash, whether F-type or C-type. Leaching studies for various trace and heavy elements were carried out using three different methodologies considering the various conditions that the CCR samples are likely to encounter (in the real world) when exposed to the environment. Such studies allow us to take decisions regarding the proper use of the ash. Thus leachate, compositional and morphological studies of thermal power waste are important as such characteristics of the materials greatly influence their use and disposal.

Pond ash samples (raw samples) from steam generating plant at the Fertilizer Corporation of India Limited, Sindri Unit were collected from four ash ponds. Samples were collected on five different days over a week and a final homogenized sample was prepared by mixing the appropriate portions. Coning and quartering were followed to prepare the homogenized sample for further studies. The

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samples were labelled as S1–S4. The SEM and elemental studies were carried (model S-415A, Hitachi Instruments Ltd, Tokyo, Japan). Loss on ignition of the samples and leaching of various elements with distilled water and under acidic conditions were also carried out.

For the experiment, 0.5 g CCR sample was taken in a conical flask and 10 ml nitric acid and 2 ml perchloric acid were added to it. Then the mixture was then heated till dryness on a hot plate in a fume hood chamber. The conical flask was covered with a funnel during the process of heating. The dried residue in the conical flask was boiled with 2 ml HNO₃ (analytical grade) and then filtered. This was repeated with distilled water, heated and filtered till no residue was left in the conical flask. The filter paper in the silica dish was kept in the muffle furnace and heated to 850°C. The silica dish was then allowed to cool with the residue in it, which was later collected for morphological analysis.

The principle of working of the SEM is that an electron beam, accelerated at 25 kV energy, is generated at the electron gun¹⁸. The electron beam is passed through various electromagnetic lenses. It first passes through condenser lens forming an electron beam spot. The fine electron beam is then focused by objective lens. The focused electron beam is deflected by beam deflection coil throughout the specimen. When the focused electron beams fall on the specimen, secondary electron beams are emitted according to the sample topography. The secondary electrons are processed to secondary electron image in the display unit through a secondary electron detector assembly.

For SEM studies, the fine powder was sonicated in a test tube with methanol solvent in a sonicator for 1 h. The fine particles were then suspended in the medium and taken out using a pipette in a cover slip and dried. After properly drying, they were subjected to gold coating to eliminate the charging effect of the electron beam during SEM observation. This was done in an ion coater (model 1132, Eiko Engineering, Japan) by sputtering technique (1400 V DC, 8–10 mA for 3 min). After gold coating, they were placed in the evacuation chamber of SEM. After evacuation, the electron beam was generated at 25 kV energy and the camera (Ashi Pentax) attached to the instrument was used to record the images.

The loss-on-ignition (LOI) is a test that measures the amount of unburned carbon in ash from coal-fired boilers. For this, 1 g of CCR sample was taken in a silica dish and heated in a muffle furnace for 1 h at 850°C. The sample was then reweighed and the loss in weight was assumed to be due to the carbon initially in the sample.

SEM can be used to determine direct compositional information about a sample through the use of EDS. The latter is either used in 'spot mode' to statically analyse a single point on the sample, or the surface is scanned to create a spatial map of elemental distribution over the sample surface. The elemental composition of the sam-

ples under study (raw and acid digested) was recorded and confirmed by EDS (model: Oxford INCA 250).

Leaching studies were conducted to know the elements that find their way into the leachates. The studies were carried out using three different methodologies considering two different conditions that the pond ash samples are likely to encounter (in the real world) when exposed to the environment. In open-column percolation experiment and 24 h shake test distilled water was used as the leaching medium. In case of acid digest test nitric acid was used as the leaching medium. The leachates collected were analysed for several elements using a flame photometer (Systronics Flame Photometer model 128 for the measurement of the sodium and potassium) and atomic absorption spectrophotometer (model GBC-902 for analysis of other elements).

As observed from the EDS study, the identified elements in the samples were Si, Al, Fe, Na, K, Mg, Ca and Ti. Table 1 shows the chemical composition of raw and digested pond ash samples. SiO₂ varied from 46.02% (S2) to 56.89% (S4) and 56.43% (S3) to 64.38% (S4) in raw and the digested samples respectively. Similarly, Al₂O₃, Fe₂O₃, K₂O, CaO, MgO and TiO₂ varied from 24.08% to 29.45% and 29.55% to 39.92%; 2.83% to 17.03% and 1.08% to 4.12%; 1.13% to 1.82% and 0.55% to 1.25%; 0.43% to 1.01% and 0.07% to 0.36%; 0.39% to 1.08% and 0.06% to 0.35%; 1.89% to 2.88% and 1.37% to 2.21% for raw and acid digested pond ash samples respectively.

From the table one can easily make out the difference in the chemical composition of the raw and the digested samples. The raw pond ash samples are typically formed of Si–Al–Fe system with traces of sodium, potassium, calcium, magnesium, sulphur and titanium. The ash is pozzolanic in nature and contains less than 7% CaO. Thus, the samples under study are class-F ash samples. The results of the digested samples showed an increase in the SiO₂ and decrease in Al₂O₃, Fe₂O₃, MgO, K₂O, CaO and Na₂O. Due to acid leaching, the alkali and alkaline earth metals were found to be leached away and thus the percentage values of components like SiO₂ and Al₂O₃ were found to increase and others such as Al₂O₃, Fe₂O₃, MgO, K₂O, CaO and Na₂O were found to decrease. Even in leaching with distilled water, the surfacial elements (alkali and alkaline earth metals) found their way into the leachates; thus, their values decreased and consequently the percentage values of SiO₂ and Al₂O₃ increased.

Pond ash also contains a variable amount of unburned carbon, depending on the combustion conditions. Unburned carbon is often measured by a LOI. In this study, LOI was determined by taking 1 g of sample (pulverized to –75 μm) in a silica dish according to standard norms. The LOI of pond ash or such materials is generally equal to the carbon content present in the sample. The unburned carbon in the samples under study varied from 9.45 to 20.33. It was highest in case of S1 (20.33%) and lowest

Table 1. Chemical composition (%) of raw and digested pond ash samples

Plant	Sample	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	Na ₂ O	K ₂ O	CaO	MgO	SO ₃	TiO ₂
Raw pond ash	S1	55.83	26.99	2.83	–	1.53	0.43	0.91	–	2.32
	S2	46.02	24.08	17.03	–	1.13	0.71	0.39	–	1.89
	S3	55.15	29.45	4.07	–	1.75	1.01	0.41	–	1.94
	S4	56.89	27.20	3.62	–	1.82	0.50	1.08	–	2.88
Acid-digested pond ash	S1	57.71	37.79	2.16	0.03	0.55	0.33	0.06	–	1.37
	S2	62.52	29.55	4.12	0.12	1.25	0.10	0.12	–	2.21
	S3	56.43	39.92	1.34	–	0.80	0.07	0.08	–	1.40
	S4	64.38	31.40	1.08	0.01	1.03	0.36	0.35	–	1.39

Table 2. Loss-on-ignition (LOI) of raw pond ash samples

Samples	LOI (%)
S1	20.33
S2	10.04
S3	9.45
S4	15.14

in case of S3 (9.45%). Unburnt carbon in pond ash is highly undesirable, as it has detrimental effects on the growth of strength of mortar and concrete. ISI has recommended that LOI should not be more than 12% for fly ash used as mortar, concrete and concrete products. High carbon content retards its pozzolanic activity. The presence of unburned carbon is the main hindrance to its beneficial application. Table 2 gives the LOI value of raw pond ash samples.

The morphological features of leached and unleached pond ash samples were examined using SEM technique to clearly understand how acid digestion affects the ash surface structure. Figures 1 and 2 show the morphology of raw and acid-digested samples respectively. Figure 1 shows that the particles are mostly spherical in shape with size varying from less than 1 to 100 μm . Some cenospheres and a few plerospheres can also be seen in the figure. Cenospheric particles show frequent bursts, which is indicative of chemical activity having occurred within them. Surfaces of some particles show extensive mechanical damage caused by impact. Small-sized particles are seen sticking to the larger spherical particles possibly on account of the convexity of the surfaces. Similarly, study of the photomicrographs of leached pond ash samples clearly shows the leaching pattern that has taken place. The particles in the leached samples lack agglomeration and are more dispersed than those in the case of unleached samples. Thus, one can conclude that the surface film or the irregularities cause the unleached particles to agglomerate.

Tables 3 and 4 provide results of leachate analysis of the four pond ash samples. Table 3 summarizes results of the leachates obtained using open column percolation

experiment. This study was done to understand the long-term leaching effects of ash when left in the pond for a longtime. Leachates from the four pond ash samples were analysed for 23 elements, and Table 3 presents the summarized results along with a comparison with IS: 2490.

Elements such as lead, chromium, nickel, cobalt, cadmium, selenium, aluminium, silver, arsenic, boron, barium, vanadium, antimony, molybdenum and mercury were all below the detection limit during the entire study period. The remaining nine elements, i.e. iron, lead, magnesium, calcium, copper, zinc, manganese, sodium and potassium were observed in the leachates. Elements like sodium, potassium, calcium, magnesium, iron, copper, zinc, manganese and lead did not show regular leaching during the period of study. Copper and zinc were within the permissible limit according to IS: 2490 in the samples. Manganese also showed initial regular leaching behaviour, then intermittent leaching and finally reduced to below the detection limit. In all the nine leached elements, high concentration was observed in the initial study period and then reduced considerably or was below the detection limit. Presence of trace elements in pond ash is a good source of nutrients in plants in agricultural applications and during reclamation of abandoned ash ponds.

Similarly, leachate analysis using 24 h shake test (Table 4) showed that the elements such as chromium, nickel, cobalt, cadmium, selenium, aluminum, silver, arsenic, boron, barium, vanadium, antimony, molybdenum and mercury were all below the detection limit. Elements that leached included iron, lead, magnesium, calcium, copper, zinc, manganese, sodium and potassium. Copper was observed only in S3. Lead was observed in S3 and S4.

The elements that leached after acid digestion included iron, lead, magnesium, calcium, copper, zinc, manganese, sodium, potassium, chromium and nickel (Table 5). Cadmium, selenium, aluminum, silver, arsenic, boron, barium, vanadium, antimony, molybdenum and mercury could not be detected. Iron was detected at higher concentrations in all the samples compared to concentrations in other test leachates. Chromium and nickel showed their presence in the leachates, which were absent in other test leachates.

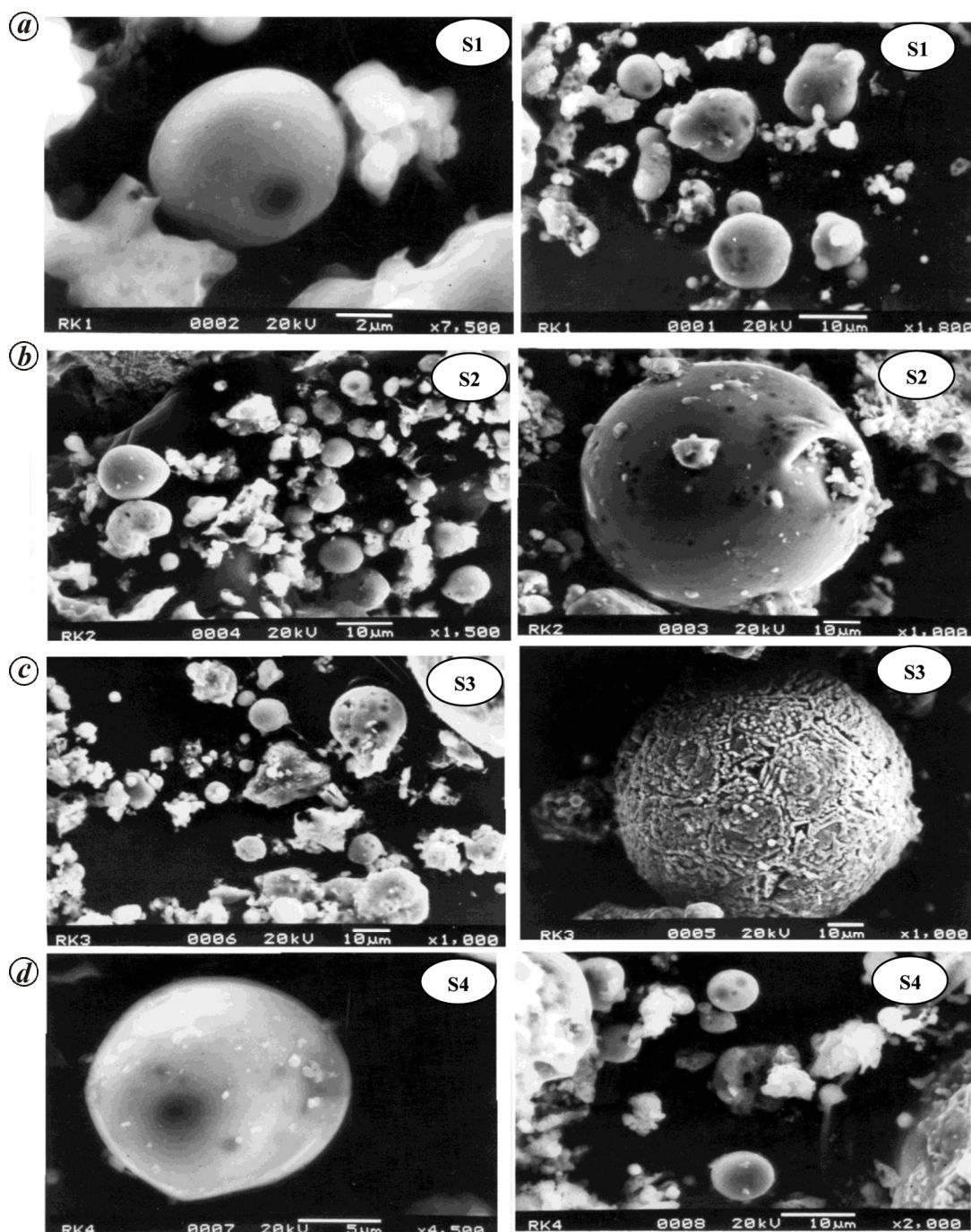


Figure 1. SEM photomicrographs of raw pond ash samples (S1, S2, S3 and S4).

Thus, the decrease in concentration of alkali and alkaline earth metals in the acid-digested pond ash as seen from the compositional studies can be better explained as the metals get leached when the ash particles come in contact with the leaching medium, be it distilled water or acid. Higher concentration and more elements find their way into the leachates when the leaching medium is changed from distilled water to acidic. Such leaching study results also support the changes in morphology of

raw and digested samples, as is evident from the photomicrographs.

The SEM-EDS observations have shown that silicon and aluminium are the major constituents. The ash samples consisted mostly of amorphous alumina-silicate spheres with lesser number of iron-rich spheres. Presence of cenospheres and plerospheres can be seen from the photomicrographs, as well as the differences between leached and unleached particles. The decrease in %

Table 3. Summary of leachate analysis (open column percolation experiments)

Parameters	S1	S2	S3	S4	(IS: 2490, 1981)	
					Inland surface water	On land for irrigation
Iron	BDL-0.900	BDL-0.522	BDL-0.800	BDL-0.760	—	—
Magnesium	0.124-86.29	BDL-20.60	0.760-50.24	0.100-54.95	—	—
Calcium	0.665-140.95	0.405-97.00	9.13-124.00	1.210-130.21	—	—
Copper	BDL-0.190	BDL-0.110	BDL-0.039	BDL-0.029	3	—
Zinc	BDL-0.384	BDL-0.106	BDL-1.391	BDL-0.155	5	—
Manganese	BDL-0.724	BDL-0.050	BDL-0.308	BDL-0.155	—	—
Sodium	BDL-18	BDL-8	BDL-18	BDL-25	—	60
Potassium	BDL-24	BDL-5	BDL-21	BDL-22	—	—

Lead, chromium, nickel, cobalt, cadmium, selenium, aluminium, silver, arsenic, boron, barium, vanadium, antimony, molybdenum and mercury are reported as below detectable limit (BDL) in the samples. Concentration of elements in ppm.

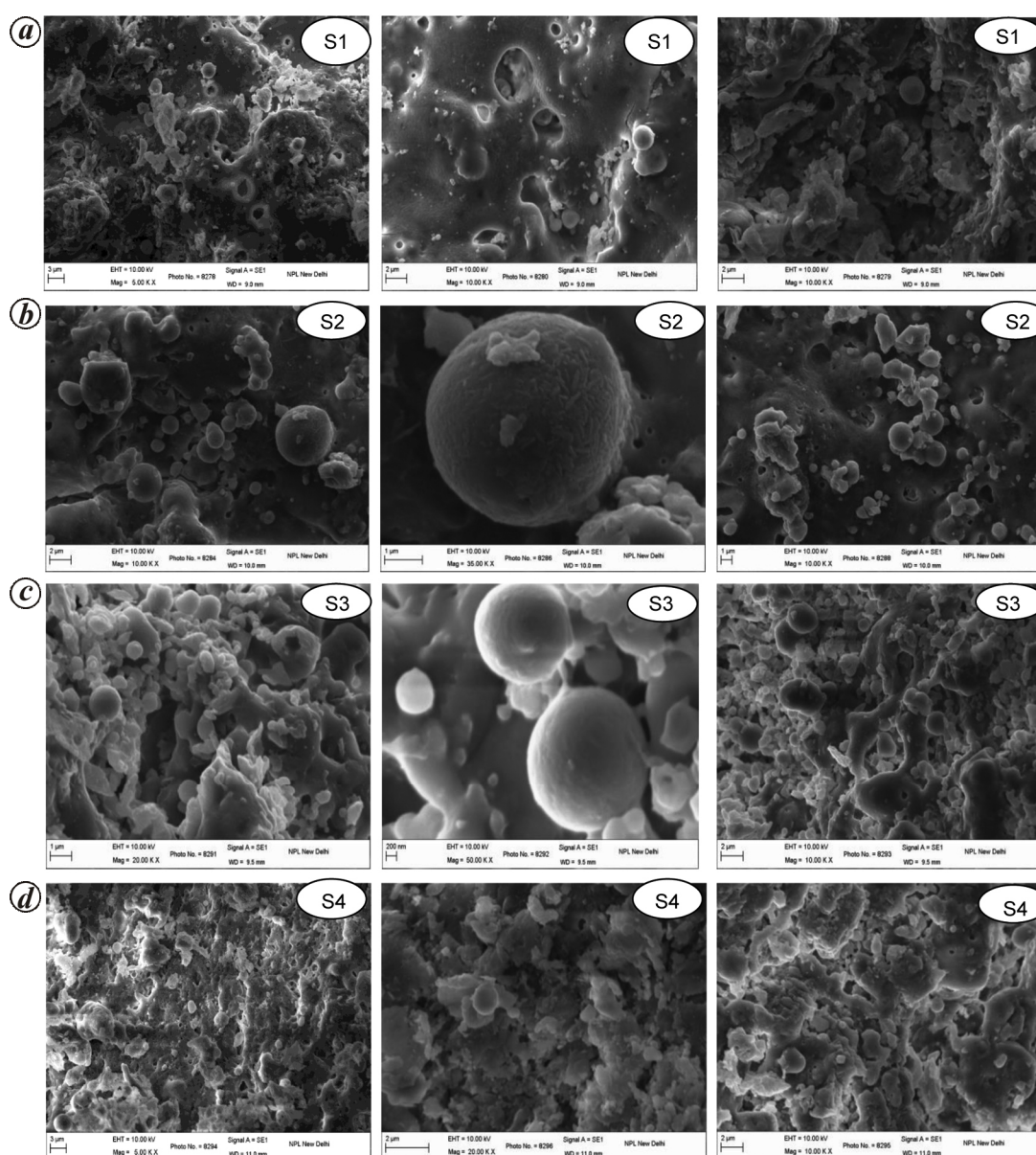
**Figure 2.** SEM photomicrographs of acid-digested pond ash samples (S1, S2, S3 and S4).

Table 4. Summary of leachate analysis (24 h shake test)

Parameters	S1	S2	S3	S4	(IS: 2490, 1981)	
					Inland surface water	On land for irrigation
Iron	BDL	BDL	0.178	BDL	–	–
Lead	BDL	BDL	0.033	0.028	0.1	–
Magnesium	1.068	0.533	1.410	1.321	–	–
Calcium	1.490	1.000	4.020	1.180	–	–
Copper	BDL	BDL	0.015	BDL	3	–
Zinc	0.022	0.055	0.063	0.037	5	–
Manganese	0.011	0.021	0.017	0.014	–	–
Sodium	4.8	7.6	5.8	6.2	–	60
Potassium	3.8	1.0	2.0	2.7	–	–

Chromium, nickel, cobalt, cadmium, selenium, aluminium, silver, arsenic, boron, barium, vanadium, antimony, molybdenum and mercury are reported as BDL in the samples. Concentration of elements in ppm.

Table 5. Summary of leachate analysis (acid digest test)

Parameters	S1	S2	S3	S4	(IS: 2490, 1981)	
					Inland surface water	On land for irrigation
Iron	9.37	162.55	238.75	16.35	–	–
Lead	0.093	BDL	0.080	BDL	0.1	–
Magnesium	423.8	7.72	10.75	6.955	–	–
Calcium	149.6	38.4	25.2	19.2	–	–
Copper	0.104	0.059	0.079	0.085	3	–
Zinc	0.112	0.135	0.127	0.236	5	–
Manganese	0.241	0.626	1.126	0.333	–	–
Sodium	5	2	5	35	–	60
Potassium	5	2	4	11	–	–
Chromium	BDL	0.214	0.372	0.845	2	–
Nickel	BDL	0.561	BDL	BDL	3	–

Cobalt, cadmium, selenium, aluminium, silver, arsenic, boron, barium, vanadium, antimony, molybdenum and mercury are reported as BDL in the samples. Concentration of elements in ppm.

concentration of alkali and alkaline earth metals oxides in the acid-digested pond ash samples has been explained through the leaching studies. The morphology of the leached pond ash samples, compositional analysis and leaching studies all corroborate.

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Assessment of hailstorm damage in wheat crop using remote sensing

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Heavy rainfall and hailstorm events occurred in major wheat-growing areas of India during February and March 2015 causing large-scale damages to the crop. An attempt was made to assess the impact of hailstorms in the states of Punjab, Haryana, Uttar Pradesh (UP), Rajasthan and Madhya Pradesh (MP) using remote sensing data. Multi-year remote sensing data from Resourcesat 2 AWiFS was used for the purpose. Wheat crop map, generated by the operational FASAL project, was used in the study. Normalized difference vegetation index (NDVI) deviation images

were generated from the NDVI images of a similar period in 2014 and 2015. This was combined with the gridded data of cumulative rainfall during the period. The logical modelling approach was used for damage classification into normal, mild, moderate and severe. It was found that the northern and southern districts in Haryana were severely affected due to rainfall/hailstorm. Eastern Rajasthan and western MP were also highly affected. Western UP was mildly affected. Crop cutting experiments (CCE) were carried out in two districts of MP. The CCE data showed that the affected fields had 7% lower yield than the unaffected fields. Empirical yield model was developed between wheat yield and NDVI using CCE data. This model was used to compute the loss in state-level wheat production. This showed that there was a reduction of 8.4% in national wheat production. The production loss estimated through this method matched with the Government estimates.

Keywords: Crop cutting experiments, hailstorm, rainfall, remote sensing, wheat.

HAIL is a solid, frozen form of precipitation that causes extensive damage to properties and growing crops¹. Hence an accurate method for monitoring and quantifying hailstorm/heavy rainfall damage to crops can be of assistance in the decision-making processes for agriculture insurance agencies and policy planners. However, current practices of hailstorm damage detection and its assessment have limitations, especially on a large spatial scale. The traditional method for damage assessment needs labour-intensive field surveys which are time-consuming with high cost. The use of remote sensing can provide alternative techniques for the assessment of damaged crop area. With the advantages of a wide swath, short repetition cycle, multiple sources and multiple resolutions², remote sensing imageries have widely been used in assessing hail damage.

An early example of hail damage detection from satellite remote sensing was the use of visible band of the GOES-8 to identify surface damage along a 120 km path of ‘almost complete vegetative defoliation and destruction’ in western South Dakota, USA that was caused by large hail (>5 cm) and severe winds (>50 m s⁻¹), including complete devastation of range grasses, planted crops and extensive defoliation of trees³. Later, remote sensing-based normalized difference vegetation index (NDVI) was used in hailstorm and wind damage assessment^{4–6}. In a study by Bentley *et al.*⁵, a hailstorm-damaged vegetation had significantly lower NDVI values as the crop was completely destroyed, whereas wind damaged vegetation had relatively minor differences in NDVI values due to only temporary disruptions in photosynthesis. Potential of infrared (IR) colour and standard colour aerial photographs was evaluated to measure the crop–hail losses⁷. Landsat TM data were used to map the hailstorm-damaged

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