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ORIGINAL RESEARCH ARTICLE

Coal combustion residues characterization using scanning electron microscopy & energy dispersive X-ray (SEM-EDXA) analysis

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ABSTRACT

The objective of the present study is to observe the surface morphology, structure and elemental composition of the ash particles produced from some thermal power stations of India using scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDXA). This information is useful to better understand the ash particles before deciding its utility in varied areas.

Keywords: Coal Combustion Residues; Characterization; XRD; SEM-EDXA; FTIR

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1. Introduction

Power sector is the major consumer of coal in our country. With rapid industrialization, since independence and improvement in the quality of life, the demand for power has increased tremendously and this has led to the increase in the consumption of coal. The increasing population has further increased the coal consumption. India is the world's third largest producer and the fourth largest consumer of electricity. More than 51% of India's commercial energy demand is met through the country's vast coal reserves[1]. The Indian power sector generated approximately 1160.141 BU of electricity in 2016-2017[2,4]. During the last two decades, the growth in the power sector has been phenomenal. Of the total power generated more than 50% has been contributed by coal-fired power station[3,4]. The electricity demand owing to increase in the population and industrialization during last one and a half decade has increased tremendously, which has led to the per-capita increase in power consumption in India to 1010 kWh in 2014-2015 and 1075 kWh in 2015-2016. The installed capacity, which was 72,320 MW in 1993-1994, has now reached 329,226 MW as on August, 2017[5,6,7].

Coal-fired power stations still dominates the energy sector in India. India has vast reserves of thermal grade coal that is cheaply and readily available as a raw material for power generation. The estimated coal reserves as on 01.04.14 in India are 301.564 BT[8,9,10]. The production of coal has increased from about 70 MT in early 1970s to 639.234 MT in 2015-2016[11]. Coal that is used for power generation in the country is mostly low-grade coal containing 30–50% ash[12,13,14]. The combustion of coal at the coal-fired power stations produces ash residues of inorganic minerals.

Around 132 thermal power stations are there in the country that meets 51% of India's commercial energy demand. These thermal power stations produce around 150MT of coal combustion residues (CCRs) per year that is causing great environmental concern in the form of air, water and land pollution besides its proper handling and disposal[3]. Disposal of such a huge amount of CCRs craves for huge tract of land besides having several environmental implications in the disposal environment. In country like India, where land resources is very limited, proper management of CCRs is the need of the hour not only in our country but also throughout the world and all these requires proper understanding of CCRs through its characterization study.

Mostly two types of ashes namely, fly ash and bottom ashes are produced by thermal power stations in India. Fraction that tries to escape along with flue gas, are trapped by electrostatic precipitators and mechanical dust collectors and is referred as fly ash. This constitutes about 80 percent of the ash produced. The other fraction collects at the bottom of the furnace and is named as the bottom ash. This is coarser in nature and constitutes about 20 percent of the ash produced. The two ashes in the wet system of disposal (as practiced in our country by most of the coal based thermal power plants) are made into slurry form and pumped to the disposal area especially made for this purpose. The ash in such disposal pond is known as pond ash.

Table 1. Status of world energy at the end of 2016[18] (BP, 2017)

	2016	**Change 2016 over 2015 (%)**
Electricity generation (TWH)	24816.4	2.2
Coal		
Reserves (MT)	1139331	-
Production (MT)	7460.4	-6.5
Production (MT of oil equivalents)	3656.4	-6.2
Consumption (MT of oil equivalents)	3732.0	-1.7

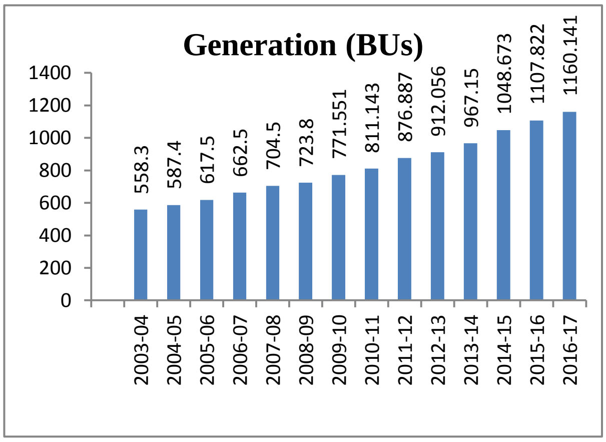
Table 2. Year-wise coal consumption, power generation and generation growth in India (2003-2004 to 2013-2014)[19,20,21,22]

Year	**Coal Consumption (Million Tonnes)**	**Generation (BUs)**	**Generation Growth (%)**
2003-2004	-	558.30	-
2004-2005	278.00	587.40	5.21
2005-2006	281.00	617.50	5.12
2006-2007	302.00	662.50	7.29
2007-2008	330.00	704.50	6.34
2008-2009	355.00	723.80	2.74
2009-2010	367.00	771.551	6.60
2010-2011	387.00	811.143	5.13
2011-2012	417.56	876.887	8.11
2012-2013	545.60	912.056	4.01
2013-2014	489.40	967.150	6.04
2014-2015	530.40	1048.673	8.43
2015-2016	545.90	1107.822	5.64
2016-2017	-	1160.141	4.72
2017-2018	-	1229.400 (Target)	5.97

The major challenge before the nation is to effectively utilize these coal combustion residues in bulk and that too in environmentally benign manner. The increasing number of thermal power stations and also the ever increasing population will add more pressure for immediate utilization. The ash utilization in India in 1992-1993 was 2–3%[15], which has now increased to 60.97% of CCRs as per the latest report[16]. However, the percentage of ash utilized is still low as compared to other developed countries of the world where utilization is close to 100%[17]. Though in our country the situation has changed since last one and a half decade, we have to go a long way to fulfill the promise of 100% utilization.

Table 1 shows the status of world energy at the end of 2016. **Table 2** and **Table 3** show the year-wise coal consumption and power generation in India from 2003-2004 to 2013-2014 and per-capita electricity consumption in India from 2005-2006 to 2015-2016, respectively. **Table 4** shows the per-capita consumption of electricity by leading countries of the world. **Table 5** shows the India's CCRs utilization scenario. Similarly, **Table 6** provides the data on CCRs utilized by the leading countries of the world. **Figure 1** shows year-wise power generation in BUs and coal consumption in MT in India.

This paper provides a detailed characterization study of CCRs from Indian thermal power station. The paper covers SEM-EDXA analysis of the CCR samples. The actual aim of this paper is to understand CCRs suitability in various applications using the characterization data so that the material can be used in an environmentally friendly manner.



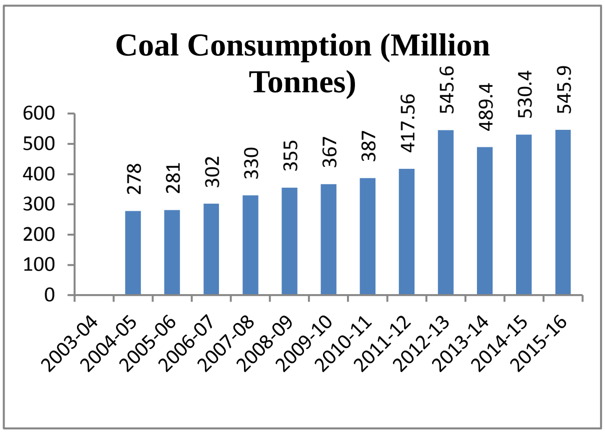


Figure 1. Year-wise power generation (BUs) and goal consumption (MT) in India**.**

Table 3. Year-wise per-capita electricity consumption in India (2005-2006 to 2015-2016)[23,24]

Year	Consumption (kWh)
2005-2006	631
2006-2007	673
2007-2008	717
2008-2009	734
2009-2010	779
2010-2011	819
2011-2012	884
2012-2013	914
2013-2014	957
2014-2015	1010
2015-2016	1075

Table 4. Comparative per-capita consumption of electricity (kWh)[25,26]

Countries	Consumption (kWh) for 2013
Canada	15520
USA	12987
Australia	10067
Japan	7836
France	7382
UK	5409
World	3026
India	957

Table 5. Year-wise CCRs utilization in India (1993-1994 to 2005-2006)

Year	Utilization (%)
1992-1993	2-3%
2002-2003	22.68
2003-2004	29.39
2004-2005	38.04
2005-2006	45.69
2006-2007	50.86
2007-2008	53.00
2008-2009	57.11
2009-2010	62.60
2010-2011	55.79
2011-2012	58.48
2012-2013	61.37
2013-2014	57.85
2014-2015	54.31

2. Materials and methods

2.1 Selection of thermal power stations

For assessing the environmental characteristics of coal combustion residues (CCRs) five thermal power stations, viz. three of Damodar Valley Corporation (DVC), namely, Bokaro Thermal Power Station (BTPS), Chandrapura Thermal Power Station (CTPS) and Durgapur Thermal Power Station (DTPS), one fluidised bed combustion (FBC), Plant of Tata Iron & Steel Company (TISCO) and one of Fertilizer Corporation of India Ltd. (FCI), Sindri unit were chosen. All the thermal power stations are either located on the banks of Damodar river or in its immediate vicinity in the states of Jharkhand and West Bengal.

These thermal power stations were chosen for the following reasons:

- These form the life-line of an industrial belt in Damodar river basin
- Power plants and the ash ponds are close to surface water bodies.
- There is probability of surface and ground water contamination due to leaching of trace elements from the ash ponds of these plants.

Also, these thermal power stations were easily accessible and it was possible to get desired facilities for the studies. **Figure 2** shows the locations of the power stations under study along river Damodar. Similarly, **Table 7** gives the composition of coal being used at different thermal power stations.

Table 6. Utilization of fly ash by various countries[15]

S.No.	Country	Utilization (%)
1	Australia	40
2	Canada	40
3	China	35
4	Czechoslovakia	40
5	Denmark	85
6	France	70
7	Germany FR	85
8	Greece	45
9	Hungary	50
10	India	41
11	Israel	80
12	Japan (a)	40
13	Netherlands	100
14	Poland	100
15	South Africa	35
16	U.K.	60
17	U.S.A	35

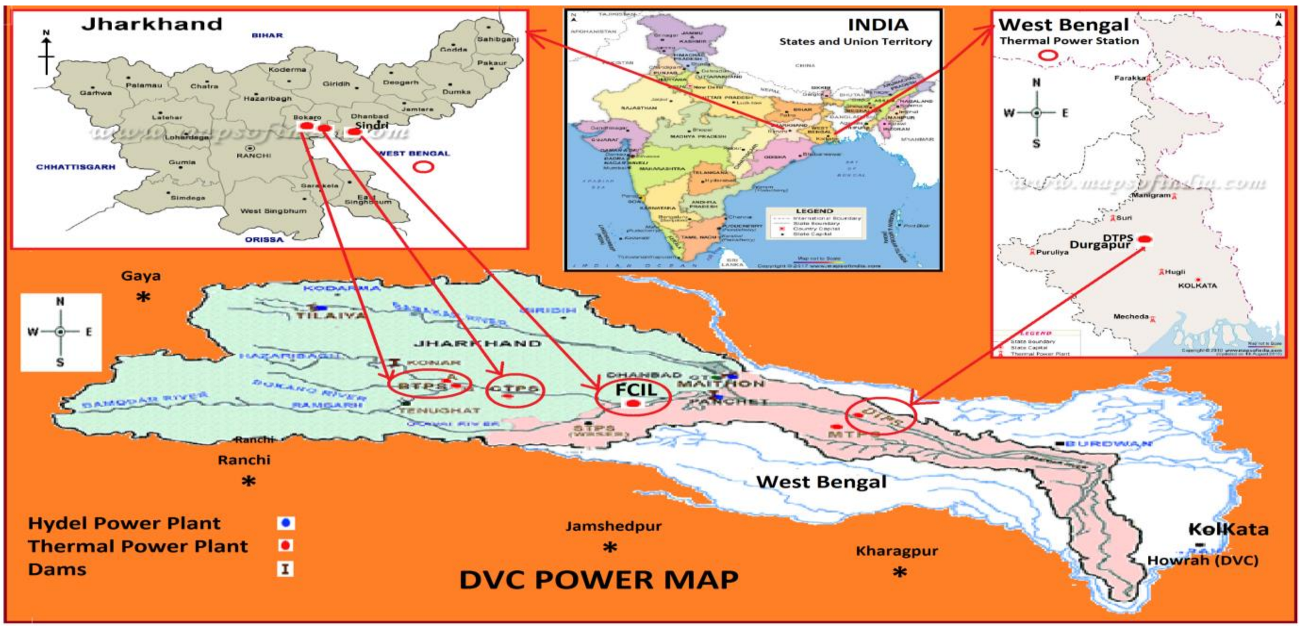


Figure 2. Location of Bokaro thermal power stations along the River Damodar.

Table 7. Composition of coal being used at different thermal power stations[27]

Parameters	BTPS	CTPS	DTPS	FBCP	FCIL
Fixed carbon (%)	36.28–47.77	41.75	55.30	26.67	45–47
Volatile matter (%)	15.8–18.12	16.20	24.50	-	16–19
Moisture (%)	0.9–1.11	0.60	2.80	1.1	0.5
Ash (%)	34.23–40.25	41.45	27.40	65	30–35
Gross calorific value (kcal/kg)	4670–4970	4665	5560	2200	4800

2.2 Sampling

Fly ash (FA) and bottom ash (BA) samples were collected on five different days over a week and a final homogenized sample for each of the fly ash and bottom ash were prepared by mixing the appropriate portions. Similarly, pond ash samples were collected from the ash ponds site from five different locations on five different days over a week and a final homogenized sample was prepared mixing appropriate portions. The CCR samples after coning and quartering method were then taken for characterization studies adopting analytical methods.

2.3 Scanning electron microscopy & energy dispersive X-ray analysis

The scanning electron microscopy (SEM)[29,30] allows high-level magnifications, which can be used for studying morphology of the sample of finer materials. It uses a focused electron beam to scan small areas of solid sample surfaces. Secondary electrons are emitted from the sample and are collected to create an area map of the secondary emissions. This secondary emission is very much dependent on the surface characteristics and so the area obtained is a magnified image of the sample. This technique is also referred to as energy dispersive X-ray analysis (EDXA). The back-scattered electrons also produce X-rays and the same can be utilized by many instruments for the qualitative compositional analysis of microscopically small portions of the sample. This technique is also referred to as energy dispersive X-ray analysis (EDXA). The sample requirement for the SEM analysis is that the solid samples, viz. thin films, powder, fibers and bulk materials should be vacuum compatible.

The principle of working of the instrument is that an electron beam, accelerated at 25 KV energy, is generated at electron gun. The electron beam is passed through various electro-magnetic 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. As soon as the focused electron beams falls on the specimen, the secondary electron beams are emitted as per the sample topography. The secondary electrons are processed to secondary electron image in the display unit through a secondary electron detector assembly.

The scanning electron microscopic studies were carried out using Model S-415A (**Figure 3**), Hitachi Instruments Ltd., Tokyo, Japan. The fine powders are sonicated in test tube with methanol solvent in a sonicator machine for 1 hour. The fine particles were then suspended in the medium. It was then pipetted out through a pipette in a cover slip and dried. After properly drying, it was 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 at a 1400V D.C., 8-10 mA current for 3 minutes. After gold coating, it was placed in the evacuation chamber of SEM. After evacuation, the electron beam was generated at 25 KV energy and the secondary electron Ashi Pentax Camera was attached to the instrument to record images and the photomicrographs were taken one by one.

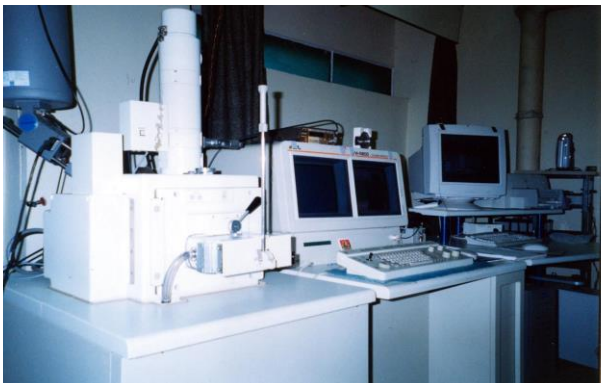


Figure 3. Scanning electron microscope, Hitachi Instruments Ltd., Japan, Model 415A**.**

2.4 Results and discussion

The results of the characterization of coal combustion residues (CCRs) from five thermal power stations, viz. BTPS, CTPS, DTPS, FBCP and FCI, Sindri for assessing their characteristics for various uses as determined by instrumentation techniques such as SEM-EDXA is discussed below.

3. SEM-EDXA

The morphological features of the leached and unleached CCR samples were examined with the help of scanning electron microscope using scanning electron microscopy (SEM) technique. **Table 8** gives the observation made with respect to SEM studies of CCR samples under study. These are also shown in **Figure 4** to **Figure 8**. The study of the micrographs of the unleached CCR samples in general indicated that CCR consisted primarily of spherical particles with nodules present on it. The particles were of different sizes and ranged from 1 µ to 100 µ. Similarly, study of the micrographs of leached CCR samples clearly shows the leaching pattern that has taken place. The particles in the leached samples lacked agglomeration and were more dispersed than one can observe in the case of unleached samples. Thus, one can conclude that the surface film or the irregularities caused the unleached particles to agglomerate.

Figure 9 shows few micrographs of the leached CCR samples. One can easily observe the leaching phenomena that have taken place and that the surfaces of the leached particles were observed to be smoother. It means that the material residing on the surface has been washed away during the leaching. Surficial element mostly present included alkali and alkaline earth metals, i.e. sodium, potassium, calcium and magnesium. As these got washed away due to the first flush phenomenon, their presence in the leached samples also decreased considerably. Decrease in concentration of these elements with time can be very well observed from the plot of the open column percolation experiment results for these elements.

Some of the particles on the leached samples were found distorted as can be seen from the micrographs of the leached samples. Distortion of particle surface is due to dissolution or disruption of the surface, making the wall thinner and thinner and finally rupturing the wall.

As is pointed out, the particles are mostly spherical in shape and they are either hollow spheres commonly known as cenospheres or solid spheres or may be containing many smaller spheres within a sphere known as plerosphere. All three can be seen from the micrographs. Cenospheres and plerospheres are present in very low amount. Some spongy morphology can also be noticed from the micrographs. A point of special importance is the fact that most of the particles are found to be of spherical nature. Due to being spherical mixed with cement, it can add workability to cement concrete mix. Being spherical and hollow can be used as filler in paints and so on.

Table 8. Summary of SEM study of CCR samples from a few thermal power stations of India

Plant	Samples	SEM (Observations)
PS	FA#A; FA#B; BA#A; BA#B; PA	• Mostly spherical in shape with size varying from less than 1 micron to 100 micron. • Particles were found mostly spherical in shape with nodules present on it. • Some cenospheres could also be seen from the micrographs. • A few plerospheres could also be seen from the micrographs. • Cenospheric particles show frequent bursts which are inductive of chemical activity having occurred within them. • Surfaces of some particles show extensive mechanical damage caused by impactation. • Small size particles were seen sticking to the larger spherical particles possibly on account of the convexity of the surfaces. • Leached particles were observed to be smoother. This shows the washout of the elements residing on the surface with time. Plot of OPCE also shows this decreasing trend. • Some spongy morphology could also be seen from the micrographs.
CTPS	FA#1; FA#2; BA#1; BA#2; PA	
DTPS	FA; BA; PA	
FBCP	FA; PA	
FCIL	S1; S2; S3; S4	

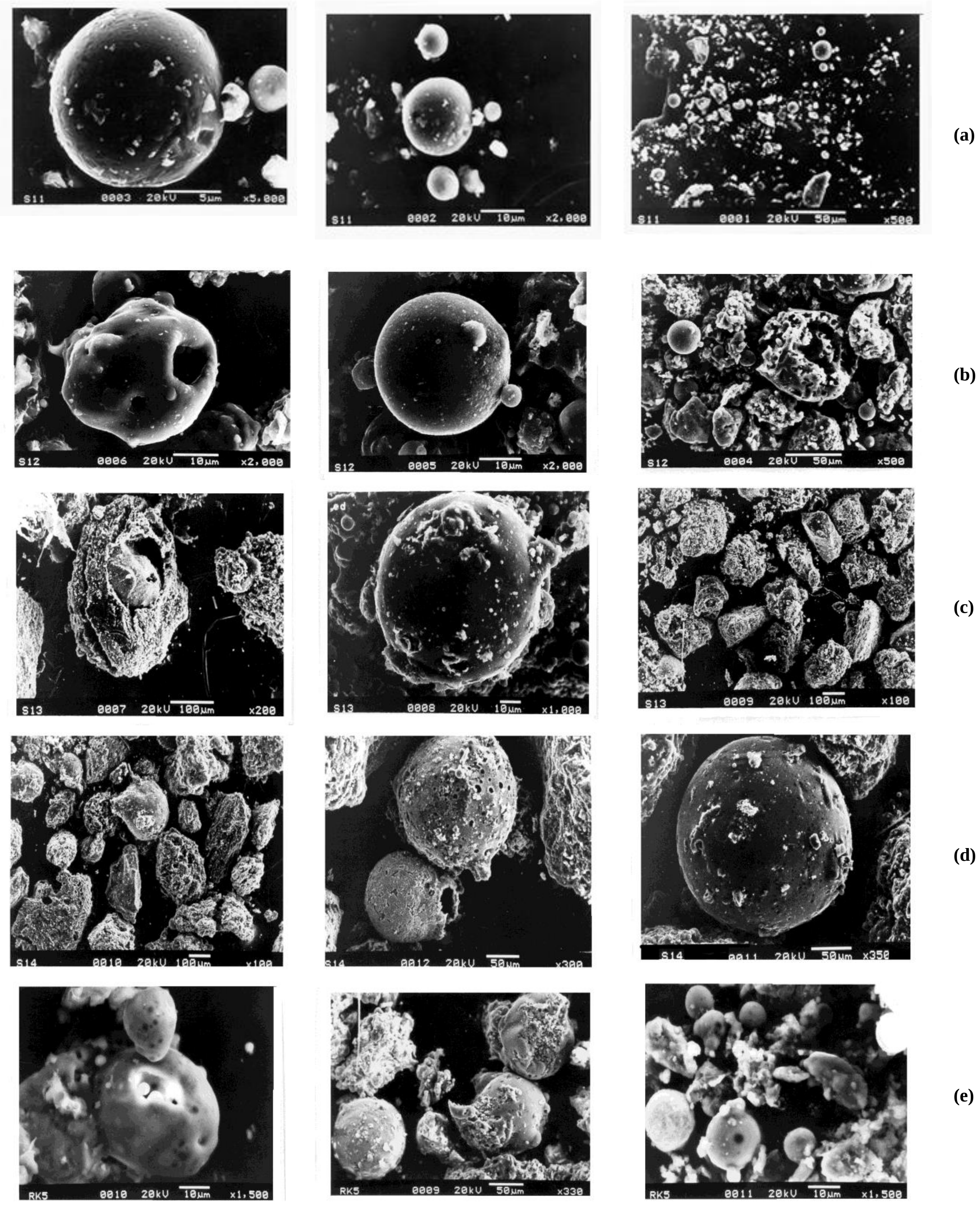


Figure 4. Scanning electron micrographs of BTPS **(a)** FA#A; **(b)** FA#B; **(c)** BA#A; **(d)** BA#B and **(e)** PA**.**

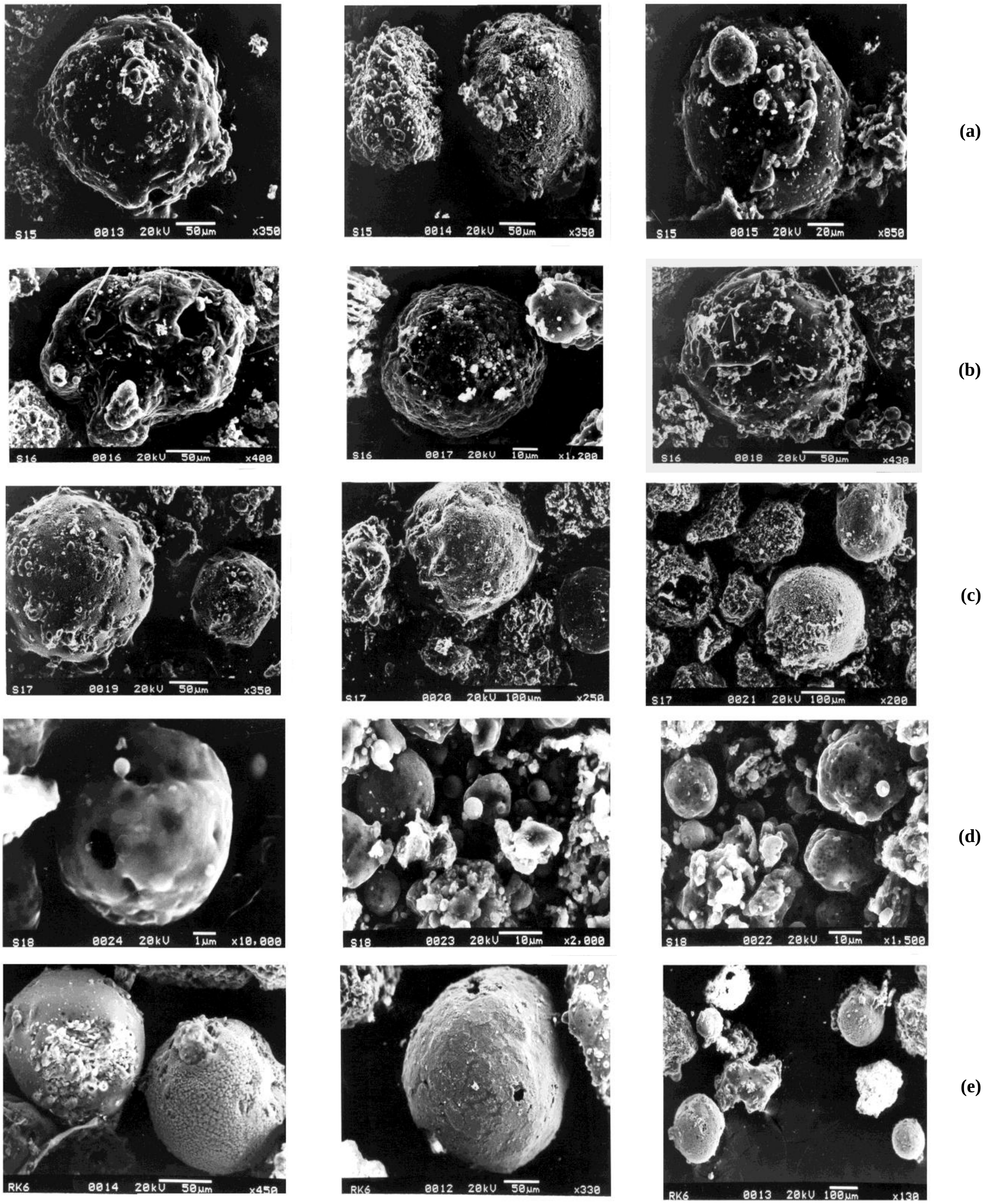
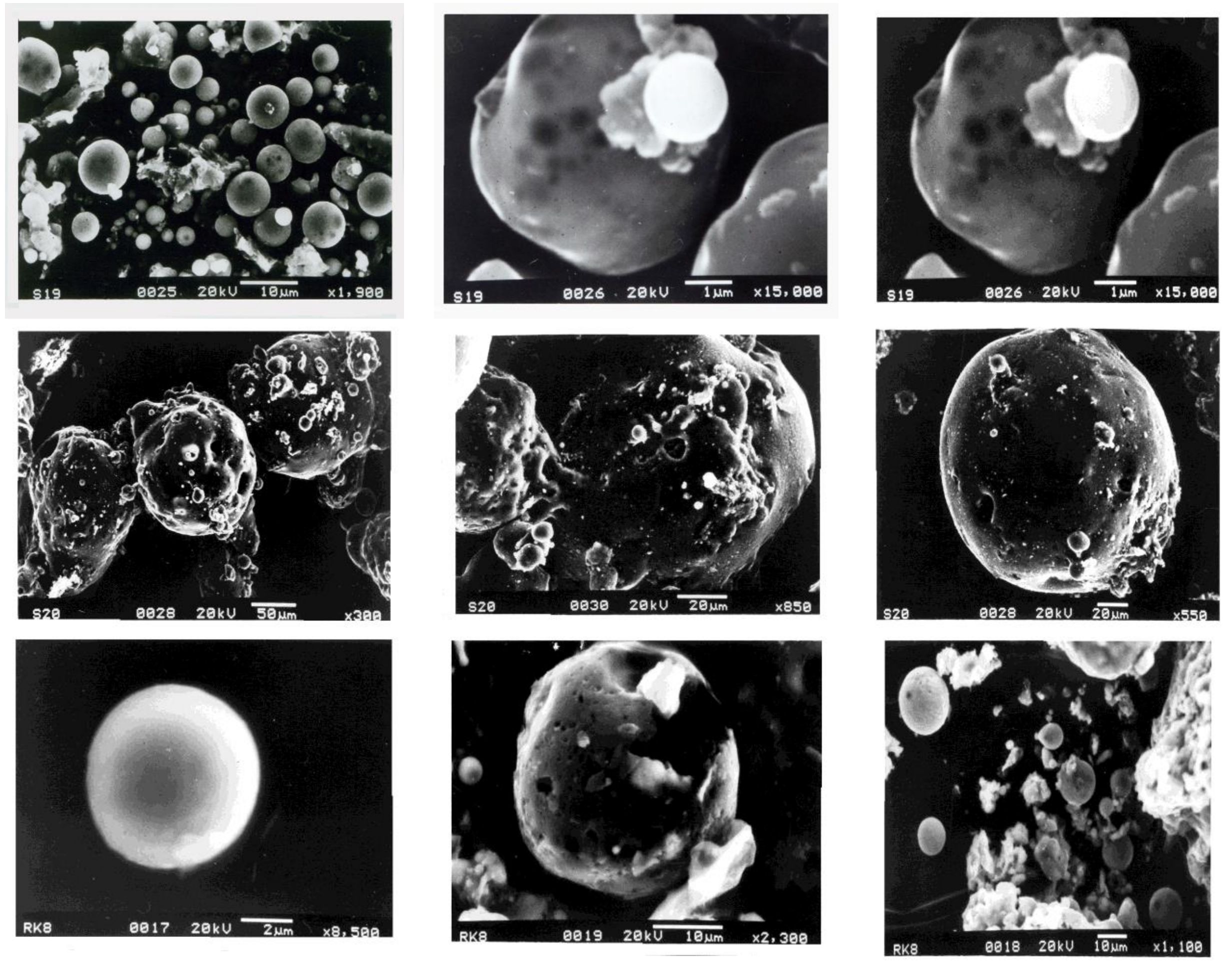


Figure 5. Scanning electron micrographs of CTPS **(a)** FA#1; **(b)** FA#2; **(c)** BA#1; **(d)** BA#2 and **(e)** PA**.**

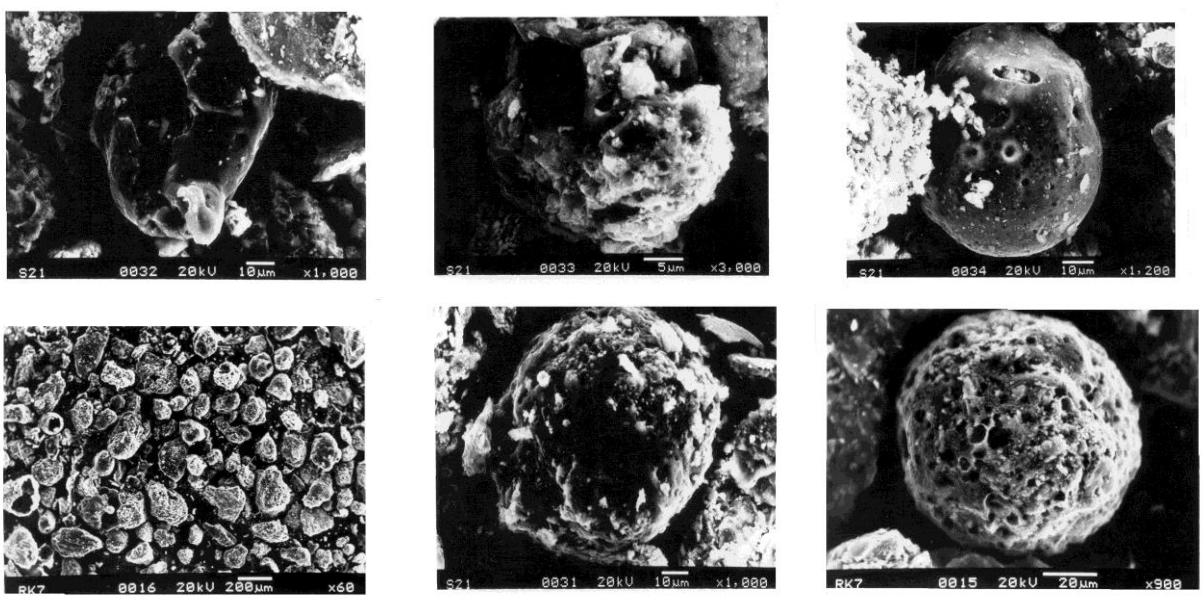


(a)

(b)

(c)

Figure 6. Scanning electron micrographs of DTPS **(a)** FA; **(b)** BA and **(c)** PA**.**



(a)

(b)

Figure 7. Scanning electron micrographs of FBCP **(a)** FA and **(b)** PA**.**

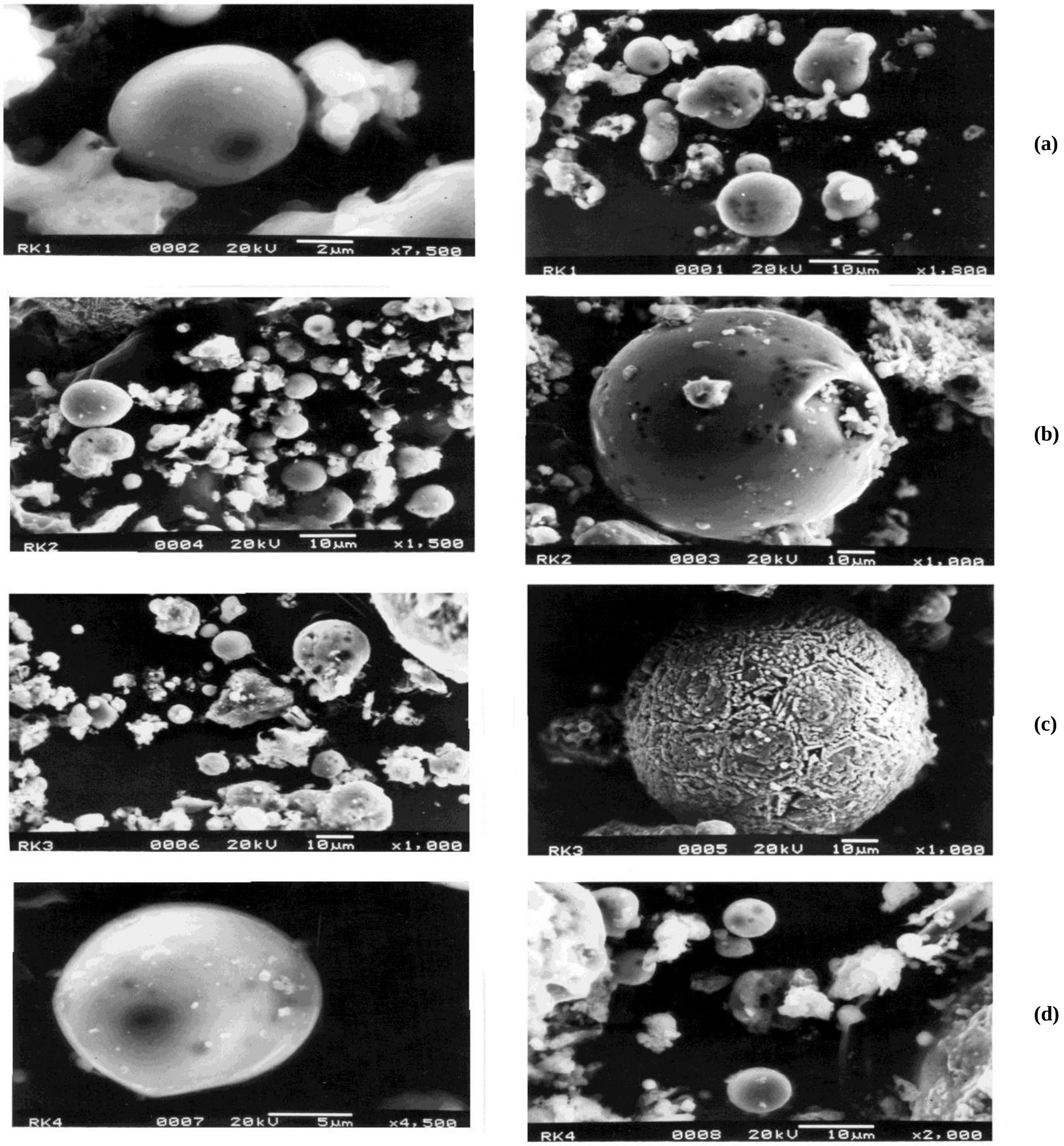


Figure 8. Scanning electron micrographs of FCI **(a)** S1; **(b)** S2; **(c)** S3 and **(d)** S4**.**

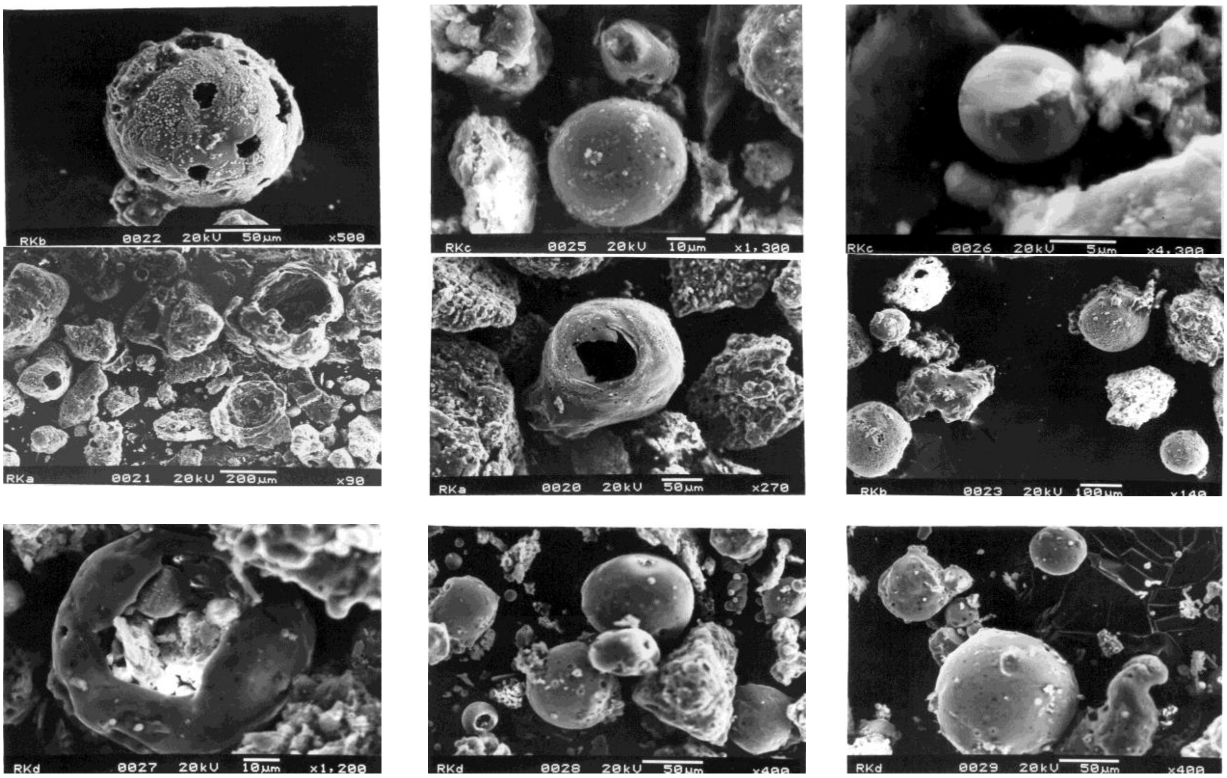


Figure 9. Scanning electron micrographs of CCR samples after leaching**.**

The observations of EDXA analysis are given in the **Table 9**. This study was performed to determine the trace element contents in the CCR samples. The study shows that the CCR samples are typically formed of Si-Al-Fe system with traces of sodium, potassium, calcium, magnesium, sulphur and titinium.

Table 9. Summary of EDXA of CCR samples from a few thermal power stations

Plant	**Samples**	**Si**	**Al**	**Fe**	**Na**	**K**	**Ca**	**Mg**	**S**	**Ti**
BTPS	FA#A	25.97	14.85	3.34	2.52	1.63	0.62	0.12	0.02	0.97
	FA#B	21.67	14.56	3.75	6.35	1.42	0.89	0.92	0.06	2.16
	BA#A	25.88	17.36	2.07	1.93	1.27	0.09	0.02	-	1.32
	BA#B	26.40	15.93	1.36	1.10	1.66	0.32	0.64	-	1.04
	PA	23.20	12.20	8.54	4.36	1.16	0.37	0.90	0.21	1.06
CTPS	FA#1	27.35	14.54	5.09	0.01	1.11	0.44	0.05	0.72	0.91
	FA#2	25.66	16.94	2.92	-	1.16	0.15	0.50	0.02	0.53
	BA#1	27.34	14.70	2.78	0.84	1.15	0.31	0.21	0.28	1.61
	BA#2	22.90	15.90	7.28	1.41	1.23	0.90	0.64	-	1.69
	PA	24.64	14.88	4.25	0.65	1.06	0.41	1.01	0.38	0.79
DTPS	FA	25.73	13.36	3.11	2.78	1.62	0.71	0.86	-	1.42
	BA	24.87	12.49	4.81	4.73	1.31	1.51	1.13	0.15	0.06
	PA	18.05	22.62	1.01	1.43	0.75	0.34	0.46	0.05	1.03
FBCP	FA	24.97	13.11	4.63	1.77	2.61	2.77	0.80	0.51	1.34
	PA	22.27	19.64	2.31	0.35	1.28	0.78	0.63	0.59	1.66
FCI	S1	26.10	14.28	1.98	-	1.27	0.31	0.55	-	1.39
	S2	21.51	13.17	11.91	-	0.94	0.50	0.23	-	1.14
	S3	25.78	15.59	2.84	-	1.45	0.72	0.25	-	1.16
	S4	26.59	14.40	2.54	-	1.26	0.35	0.65	-	1.73

4. Conclusion

The SEM studies of the CCRs have shown that CCRs consisted primarily of spherical particles with nodules present on it. The particles varied in size from less than 1 micron to 100 micron. The particles consisted of cenospheres (hollow spheres), solid spheres and plerospheres (spheres within a sphere). Spongy morphology and a small amount of angular shaped particles were also seen. This was due to the presence of unburned carbon and other minerals. Being spherical and hollow these particles can be used as filler in paints and so on. On the other hand, the SEM studies of the CCR particles after leaching have clearly shown the leaching pattern that has taken place. The particles after leaching lacked agglomeration compared to the original particles. Some of the particles were also observed distorted in shape while walls of some of the particles were found ruptured. This distortion and rupturing may be accounted due to the dissolution of the surficial elements making the wall of the sphere thinner and thinner and finally the wall ruptures.

List of Abbreviations

CCRs	Coal Combustion Residues
DTPS	Durgapur Thermal Power Station
BTPS	Bokaro Thermal Power Station
CTPS	Chandrapura Thermal Power Station
TISCO	Plant of Tata Iron & Steel Company
FCIL	Fertilizer Corporation of India Limited
DVC	Damodar Valley Corporation
SEM	Scanning Electron Microscopy
EDXA	Energy Dispersive X-Ray Analysis
BP	British Petroleum
CEA	Central Electricity Authority
TEDDY	Teri Energy Data Directory and Yearbook

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