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Forensic Investigation of Coal Comparison by Conventional and Modern Analytical Technique: A Case Study

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Abstract: Coal is the world's largest source of energy for production of electricity. Coal samples seized under section 406, 420 of Indian Penal Code (for criminal breach, cheating and dishonesty) were sent for forensic examination. Forensic investigations which include comparison and characterization of coal samples showed that poor quality coal samples are rich in moisture and ash content as compared to genuine coal samples. The micro X-ray fluorescence analysis showed high values of minerals in suspected samples as compared to genuine coal. Thus the results of proximate analyses of coal are in good agreement with the results obtained by advanced elemental detection techniques and hence provide valid evidence in cases of cheating or adulteration.

Keywords - Ash content, Coal comparison, Elemental detection, Forensic Examination.

1. Introduction

Coal, also called as Black Gold, is world's largest source of energy for the production of electricity. In India it fulfills almost 67% of total commercial energy consumed. This fossil fuel is widely used both for domestic as well as industrial purposes. Coal is largely made up of carbon. It also contains mineral matter as well as hydrogen, oxygen, sulphur and nitrogen. Generally coal is classified in different types depending on its carbon content viz. peat (40%), lignite (40-55%), bituminous (60-80%) and anthracite (80-95%). The analytical assessment of properties such as volatile matter, moisture, fixed carbon, ash content and calorific value are very important to decide the quality of the coal[1] and the utility of coal depends on the properties of coal.

These different types of coal vary in chemical, physical, thermal and plastic properties. Various tests are available for determination of properties of coal. Characterization of coal is carried out using proximate and ultimate analysis[1-4]. The characterization of coal is defined by certain physical parameters such as moisture, volatile matter, ash and fixed carbon and these properties are studied under proximate analysis[1-5]. The ultimate analyses of coal include determination of carbon, hydrogen, nitrogen, sulphur and oxygen. These results are essential for industrial purposes where coal is the main source of fuel. Among different types of characterizations available for coal, proximate analysis is the easiest and can be carried out at plant level with minimum infrastructure.[3]

Moisture content is the one of the important parameters in proximate analysis of coal and it is defined by the type or source of the water content in the coal. Surface moisture is water held by surface of the coal, water held by decomposed organic compounds is decomposition moisture, and water held by the part of the crystal structure of hydrous silicate such as clay is called mineral moisture however the moisture that is trapped by the capillary action within the micro fracture of the coal is called hygroscopic moisture. Groundwater and other extraneous moisture are readily evaporated and are known as adventitious moisture. Inherent moisture is moisture held within the coal and is generally analyzed quantitatively[2].

Ash content of coal is also another important parameter in coal examination. It is the incombustible material remaining after combustion of coal. High ash content in coal affects the calorific value of coal and hence the important parameter from industrial utilization point of view. Similarly the study of trace elements in coal ash is crucial for the potential impact on the geoenvironmental from its disposal and utilization[6]. Metals present in the coal ash cause various health implications on the human body. The literature for the analysis of coal ash for trace elements focused on environmental concern as well as for industrial purposes[7-10]. Coal analysis mainly focused on conventional techniques. Modern techniques such as X- ray fluorescence, AAS and ICP methods have been used for element detection in coal.[11-12]. The advanced operator independent and faster computer-controlled scanning electron microscopy (CCSEM) was used for determination of particle size, shape factor, and elemental composition of coal utilization[8]. Analysis of coal is very essential for determination of calorific value in power plants.

In the present forensic investigation for coal comparison, proximate analyses and characterization of seized coal samples were performed in detail and results obtained by classical method were compared with the advanced instrumental technique.

2.0 Case history

A coal supplier got a tender to supply coal from mine to a company. Three trucks were loaded with good quality coal from mine. Coal samples were exchanged at the coal plots of the accused where good quality coal samples were unloaded and poor quality coal samples were reloaded into the truck and were sent to the company. The company owner, doubting the quality of coal, lodged a complaint against the coal supplier for cheating under IPC 406, 420. Original coal samples from mine and exchanged coal samples were submitted to the forensic science laboratory for comparison and the details of the exhibits received are as follows-

Exhibit No.1- Coal sample from mine

Exhibit No.2- Coal loaded from accused coal plot in Truck no.1

Exhibit No.3- Coal loaded from accused coal plot in Truck no.2

Exhibit No.4- Coal loaded from accused coal plot in Truck no.3

Exhibit No.5-Coal from mine unloaded to accused site

Exhibit No.6- Coal collected from Accused coal plot

The aim of forensic investigation in this study was to compare the properties of coal samples provided by the complainant with the coal samples seized from crime scene or from accused. Hence the investigation regarding the comparison of original coal samples to substituted poor quality coal samples was carried out on the basis of conventional and instrumental method of analysis.

3.0 Experimental

All chemicals used for analysis are AR grade. Proximate analysis of coal was carried out using the standard method of analysis[1].

3.1 Moisture content- Moisture content in the coal was determined by using Hexatech oven model no. 023. About 5 grams of finely powdered coal sample was kept in a silica crucible and heated in an oven at 105^0-110^0C for one hour. The crucible was taken out, cooled in desiccators and the sample was weighed. The moisture content in the exhibit was given as follows:

% Moisture content in coal= (loss in weight of coal X 100)/weight of coal taken for analysis

3.2 Ash content- After analysis of moisture content, crucibles containing exhibits were kept in a muffle furnace at 750^0C for about 2 hours, till constant weight was achieved. Ash content of the exhibit was found out using Hexatech muffle furnace model no. 022. Ash content in exhibit was calculated as-

% Ash content=weight of residue ash formed X 100/weight of coal (initially taken for moisture determination)

3.3 Elemental analysis- The element detection was carried out on Horiba micro XRF XGT 7200. Samples of coal ash in exhibits were used for Elemental analysis. Elemental XRF (XGT) point analysis was performed at conditions:- XGT diameter: 1.2 mm; X ray tube voltage: 50 KV; X ray filter : None ;Current: 1 mA. The analysis was performed for 100 seconds under partial vacuum. The results of the analysis were given as the average abundance of the elements. The results for the values for mineral matter and specific gravity were calculated according to standard Indian coal analytical method[1].

4.0 Results and discussion

Prima facie, coal in exhibits no. 1 and 5 are hard, black and uniform in appearance while coal in exhibits no. 2, 3, 4 and 6 are black brown and brittle lumps. The morphological study indicate that the exhibits are different in their properties and hence the differences under proximate analysis are studied as follows-

4.1 Moisture content: Normally, moisture content in the coal varies from site to site. It also varies with type of coal. The comparison of moisture content of the actual coal to be transported to the one seized from crime scene coal samples were carried out. The results of moisture content are presented in **table 1**. Exhibit No. 1 and 5 show resemblances in values of percent moisture content (about 9%) however Exhibit No. 2, 3, 4 and 6 show comparable values for the same parameters (12-15%). Higher the moisture content, lower is the efficiency of coal. The high values of moisture in exhibit no. 2, 3, 4 and 6 clearly indicate poor quality of coal samples as compared to the coal in exhibit no. 1 and 5.

4.2 Ash and mineral matter: The amount of coal ash is determined by the content of the basic inorganic elements, such as silica, strontium, iron and calcium. These properties reflect the quality of coal which forms the basis for comparison. On heating exhibits in muffle furnace for ash determination, off white to pink colored ash was observed for exhibits no. 1 and 5 while brick red coloured ash was found for exhibits no. 2, 3, 4 and 6. Ash content is found to be 23.5% and 16.32 % in exhibits no. 1 and 5 respectively while about 62-66 % ash content was found for exhibits no. 2, 3, 4 and 6 as shown in **table 1**

Mineral matter (MM) in exhibits were calculated as follows-

MM=1.1 X ash + 0.555

But for Indian coals, the formula is-

MM=1.1 X ash

Substituting the values of ash from the table, the mineral matter in ex no. 1 to 6 are found to be, 26, 69, 75.5, 75, 18 and 73 respectively.

Exhibit nos. 2, 3, 4 and 6 contain black and brown coloured lumps of coal. Thus high value of ash and mineral matter in those exhibits are attributed to the presence of earth containing high mineral matter. Coal containing high ash is harder and stronger with low calorific value, clinker forming and has the property of producing slag in high amounts. The results indicate that the ash content of the coal is directly proportional to its density and inversely proportional to its heat value. Coal ash is formed from the inorganic components contained in coal, which can cause critical troubles, including slagging, fouling, bed agglomeration, corrosion, erosion, etc [6].

4.3 Specific gravity: The specific gravity of coal is influenced by the proportion and nature of both organic mass and mineral matter. For the same type of coal, higher the ash content, higher is the value for specific gravity. The specific gravity of bituminous coals varies between, 1.27-1.45. The following formula is valid for many coals in India[1, 5].

S= k +A/100

S= specific gravity of coal

A = % ash content

K = a constant value 1.25 (formula assumes an overall specific gravity of 1.25 for mineral matter of coal).

The values obtained using the formula are presented in table 1 and it was found that, specific gravity of coal samples in exhibit no. 1 and 5 are in the range of Indian coal (1.45), however higher values for remaining exhibits indicate some other matter like earth. The data in table 1 show that as the ash content of the coal increases so does the density of the coal. Thus results are helpful in finding out nature, type as well as quality of coal samples.

4.4 Elemental analysis : The results of elemental analysis are presented in **table 2** indicate that exhibit nos.1 to 6 contain most abundant common elements such as aluminum (Al), and silicon (Si), while sulphur (S), iron(Fe), potassium(K), calcium(Ca) and titanium(Ti) in moderate and vanadium(V) present in trace quantities. Iron content is high in ex. no. 2, 3, 4, and 6 while sulphur content is high in ex. no. 1 and 5. Traces of other elements such as phosphorus, nickel, zirconium and zinc are detected in exhibit no. 2, 3, 4 and 6 only; however exhibit nos. 1 and 5 contain traces of chromium. The results obtained by micro XRf analysis are presented in **figure 1**. Literature also supports distribution of 10 major inorganic elements, Si, Al, Ca, Mg, Fe, S, Ti, Na, P and K in coal[7].

Low content of iron and very low content of magnesium, calcium and potassium suggest no or traces of scale formation and deposit in boiler pipe tubing during combustion or in coal conversation process. Thus high ash content, mineral matter content in ex no. 2, 3, 4 and 6 are well supported by the micro XRF data. Similarly high values of specific gravity for ex. no. 2, 3, 4, and 6 may be attributed to high iron content and mineral matter. The values obtained for studying physical parameters of exhibit nos. (1) and (5) in the present paper are in good agreement with the literature for Indian coal samples.

Elemental analysis by **X**-ray fluorescence technique is found to be a useful tool in the forensic investigation for comparison of quality of coal. Many advanced techniques such as AAS, ICP and spectrophotometric methods satisfy this challenge to greater extent but above all, micro-XRF technique is a rapid, non-destructive modern technique that does not require any sample preparation.

Conclusion:

Results of forensic investigations are the main evidence in solving many crime cases. Elemental analysis by X-ray fluorescence technique is a powerful tool in the forensic investigation for comparison of coal. The present study not only compares the properties of genuine and fake coal samples but also reflects the quality of coal too. The proximate analyses as a conventional method of analysis of coal supported by advanced or modern elemental detection techniques thus provide valid evidence in cases of cheating or adulteration in forensic examinations.

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Conflict of Interest: The authors have no conflicts of interest to declare

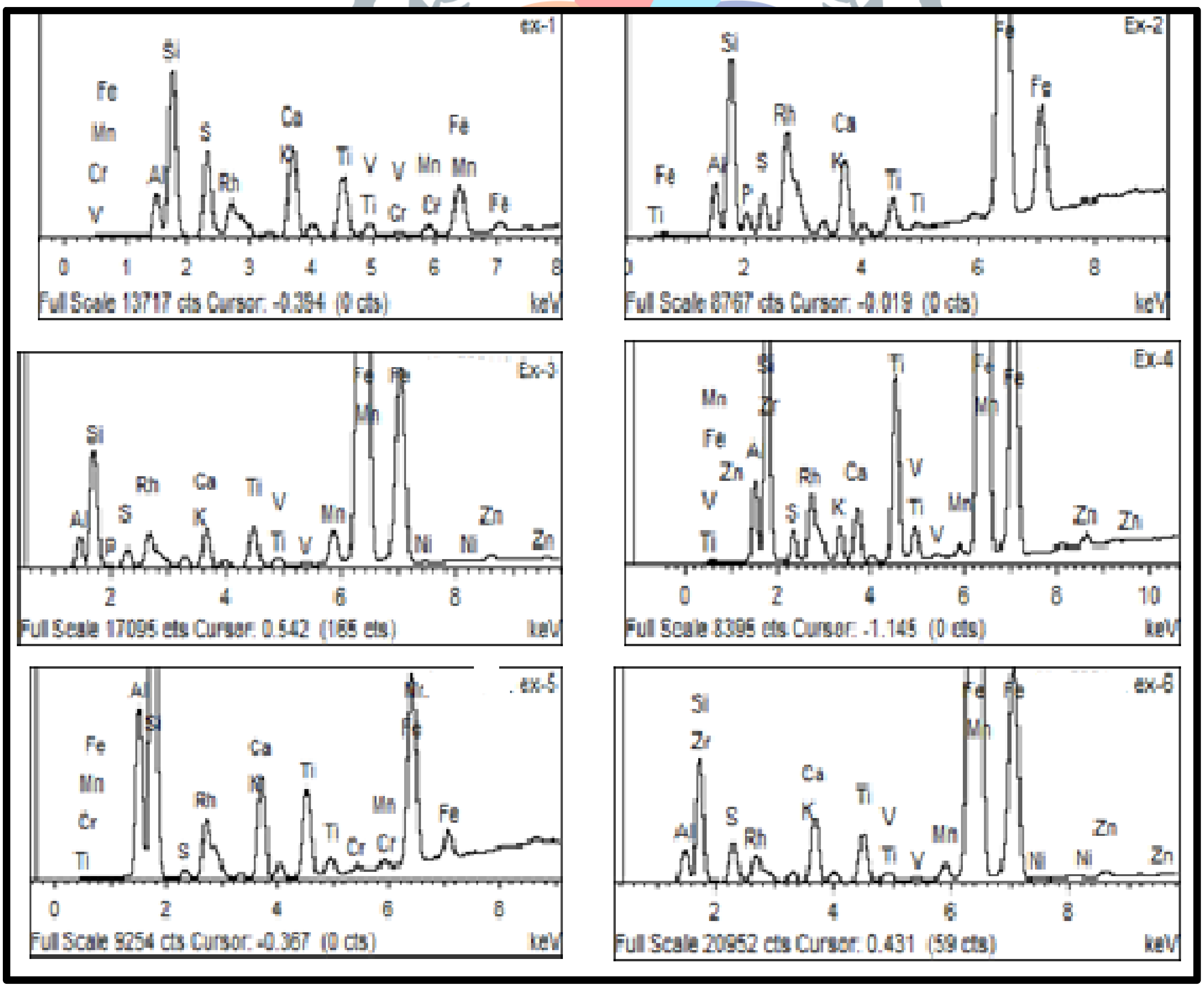
Table 1- Characteristics of Exhibits

Parameters	Exhibit 1	Exhibit 2	Exhibit 3	Exhibit 4	Exhibit 5	Exhibit 6
Appearance	Black coloured hard lumps	Black and brown coloured brittle lumps	Black and brown coloured brittle lumps	Black and brown coloured brittle lumps	Black coloured hard lumps	Black and brown coloured brittle lumps
pH	About 7 to 7.2	About 7 to 7.8	About 7 to 7.8	About 7 to 7.8	About 7 to 7.2	About 7 to 7.8
(CO_3^{2-})	Negative	++	++	++	Negative	++
Effect of dil. HCl	No coloration	Yellowish Greenish coloration	Yellowish Greenish coloration	Yellowish Greenish coloration	No coloration	Yellowish Greenish coloration
Fe	+	+++	+++	+++	+	+++
Moisture (%)	9	12	14	12	9	15
Ash (%)	23.50	62.7	68.6	68	16.32	66.5
Colour of ash	Off white to pinkish	Brick red	Brick red	Brick red	Off white to pinkish	Brick red
Mineral matter	25.85	69	75.5	75	18	73.15
Specific gravity	1.48	1.9	1.93	1.93	1.41	1.91

Table 2 Elemental composition of exhibits by micro XRF(XGT7200)

% mass	Si	Al	S	Ca	Fe	Mn	Ti	V	K	Cr	Zn	P
Ex-1	56.41	19.83	12.87	6.25	0.78	0.05	2.85	-	0.54	0.04	-	-
Ex-2	51.53	21.93	5.07	3.95	9.17	0.26	1.08	0.07	0.98	-	0.13	5.97
Ex-3	48.61	21.27	2.91	2.30	20.56	0.61	1.49	0.04	0.98	-	0.08	0.79
Ex-4	58.24	19.76	1.91	1.31	14.00	0.08	2.81	0.05	1.44	-	0.05	-
Ex-5	66.35	27.87	0.52	2.58	0.99	0.04	1.36	-	0.23	0.05	-	-
Ex-6	46.55	20.32	5.21	3.61	21.06	0.26	1.68	0.04	0.95	-	0.06	-

Figure 1-Elemental composition of coal samples in different exhibits by micro XRF



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