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Studies on the characterization of Indian coals using FE-SEM and EDS and its relevance to coal washability

The basic objective of coal washability is to establish the set of maximum possible separation performance for a given coal feed and to access the cut gravity for achieving clean coal of desired quality. The conventional float-sink (F-S) analysis is time taking, hazardous and at the same time costly. Indian coals are of drift origin and vary widely in the washability characteristics and there is also wide variation from seam to seam at the same mine. The washery is to be fed with coals from multiple sources/seams, which are having wide washability characteristics. The standard practice of washability will not help the washery to improve its performance as a result an alternative online analyzer especially for a commercial coal washing plants which maximize the reliability of actual coal washability in conjunction with particle size distribution, ash, etc is the need of the hour.

Researchers worldwide, are trying to establish the washability characteristics of coal through different routes X-ray/gamma, basically imaged based/processing to solve the problem. An automated coal washability analyser is being under developmental stage which will give near real-time F-S data that would maximize the yield at desired ash level and impact positively on the profitability of the plant operation. The results are very promising with the actual standard float and sink data to the coal analysed with the X-ray analyser, which are being highlighted in the present paper.

Introduction

Laboratory float and sink test, the washability analysis is the nitty-gritty of most of all coal preparation plant. Till date it follows conventional float and sink method, which is a time taking cumbersome task, and more over the running plants cannot be assessed instantly, ultimately the desired quality is still a challenge for coal preparation engineers. The challenges become more intense with the drifted origin Gondwana coal. Unfortunately, there are no online techniques for determining this most fundamental aspect of coal cleaning information. With the advancement of technologies it appears possible to determine coal washability online using X-ray techniques. The successful development of such a device is critical to the establishment of process control and automated coal washing/blending systems.

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Since discovery of X-rays in 1895, among the numerous important uses it has also been applied to examine the coal. First investigation reported was H. Couriot, who, in 1898, submitted anthracite, bituminous coal and other fuels to X-rays and obtained in their radiographs nearly every detail of the intimate structure of the mineral matter. Later on Mahadevan in 1929, followed by J. Dhar and B. B. Niyogi, 1942, have X-ray studied on Indian coal macerals and minerals.

In 1970's, 3D X-ray has been introduced and since advancement of X-ray, 3D structure and mineral study were carried out. The coal washability was attempted in 1990's by applying X-ray. It was reported to construct yield gravity curve using X-ray in 2000. Further in 2010, some researchers like C. L. Lin, J. D. Miller, G. H. Luttrell, G. T. Adel (USA) Gianni Schena, Luca Santoro, Stefano Favretto, Jan F. Bachmann, Claus C. Bachmann, Michael P. Cipold, Helge B. Wurst, Hauke Springer (Germany) and Mel J. Laurila (USA) were working on washability curve and later on in 2011, Germany, South Afirica, USA, etc. trying for online washability analyser. In the year 2012, S. Shamaila, B. Ntsoelengoe, J. Bachmann, H. Wurst and M. Cipold; had developed a prototype X-ray transmission washability monitor.

Atkinson, B. and Swanson, A. (2016) are working on further development in washability prediction using coal grain analysis to arrive washability of the coal using X-ray. At the same time Jan Batchman and Michael Cipold are working on online washability analyser comprising dual and triple parameter analysis. Albert Klein, Sven Reuter and Audy Zein are working on rapid coal analysis with the online X-ray elemental analyser. The researchers are working for online coal washability analyser, coal online XRF and PSD analyzer with realization of its importance.

The literature on online washability is very widely dispersed. The basic issue is that of accuracy of the online system vis-à-vis the standard F&S method, a few of the studies in this respect may be read as highlight. An important set of information in this regard is the forecast by K.P. Galvin (2006), 'Options for Washability Analysis of Coal A Literature Review', Journal *Coal Preparation*, Volume 26, Issue 4, p 209-236, 2006 that the existing Australian Standard for float and sink analysis of coal is likely to be phased out over the next five years because of its reliance on toxic organic liquids. Thus, the Australian coal industry needs to urgently find a suitable alternative, allowing enough time to establish a new approach with a new standard. It is possible that the industry in other countries will follow this lead. Several alternatives to current washability analysis exist but each of them has technical or economical disadvantages.

Hongping Liu et al have contended that there is a good match in particle size distribution and coal washability for all lithotypes in the coarser size segments. When fragmentation takes place due to shatter tests, the dull coals have lesser fragmentation and hence poorer liberation characteristics which leads to very low percentage of small particles. Discrepancies in synchronizations between washability tests and F&S tests usually differ due to smaller particle sizes and higher density material.

Miller, J. D. and Lin, C. L. used X-ray tomography to determine the washability curve of fine coal particles in different density levels. As the curve below indicates, there is good confluence between X-ray and F&S methods at higher density levels, viz. till 1.7 gms/cm^3 but then computed tomography (CT) scan method consistently depicts higher yields than F&S methods as we approach lower density levels. However, there is no corresponding data on coarser size fractions were discussed.

The laboratory float and sink tests data and the analyser readings do not match due to variations in the constituents of ash that causes density differentials even while ash content remains at relatively higher levels. It realised that < 20% ash in floats at <1.4 specific gravity is due to this phenomenon. However, since the primary objective of washing is to determine at level of washing needs to be done in order to arrive at the objective of clean coal with a specific percentage of cleans which will optimise percentage ash level as well as yield.

The levels of accuracy and prediction capabilities can be further improved by a number of methods including introducing better collimation techniques, filters, calibration with larger number of data, better correction algorithms etc.

A study by Zhang Ze-lin, Yang Jian-guo, Wang Yu-ling, Xia Wen-Cheng, Ling Xiang-yang; on "Fast Predicting the Washability Curve of Coal" published in Procedia Environmental Sciences 11 (2011) 1580-1584, wherein the accuracy levels indicated for a coal sample in size range of 13-25 mm has reached 2.375% while the China coal washing standards specify <2%. As per the claim they are quite close to the required accuracy. However, the same experiment has not been done on other types of coals and in different size ranges.

Qian Zhu in his book "Coal sampling and analysis standards" published by IEA Clean Coal Center, April 2014 has discussed in page 100-105 about the gamma source of analysis with varying composition of mineral, the ash per cent may vary greatly. The main advantages of a DUET ash gauge is its ability to measure the ash content of coal directly on a conveyor belt independently of the mass of coal on the belt and of the belt speed, but applicable to entire mass of coal on the conveyor.

Jan Bachmann, in his article, 'Online Washability: Comparison of Dual Parameter and Triple Parameter Analysis', presented at the XVIII International Coal Preparation Congress, 2016, pp 255-260 argues that dual parameters lead to high inaccuracies with changing ash content in coal as has been the experience with dual energy ash gauges. Hence, he proposes a triple parameter using an optical measurement technique to compensate for the changing variation of the third dimension in the particle. How this third dimension is going to be measured on the underside is not discussed in his article.

In the framework done by the researchers it appears that all though there is a lot of work has been carried out for instantaneous determination of ash and weight/mass in relation to density determination. The works are indicative of urgent requirements of time, comprising accuracy and speed of the results.

The development of a coal washability analyser needs the following characteristics of the coal to be measured instantaneously, i.e.:

- particle size distribution
- ash content and
- washability curves

The system must be reliable, work in an online fashion, working with minimal intervention of personnel rather to say automated with an operator, which provide nearly real-time analysis. Further, most of the analysis method is at > 6 mm while, most of the thermal coals in India, focus on >13 mm coals, and more relevant for Indian conditions of coal washing.

Technical realisation

A proto type of the coal washability is designed for use in the laboratory and was developed at CSIR-CIMFR in association with Ardee Hi-Tech Pvt Ltd as depicted in the Fig.1.

Fig.1 Laboratory model coal washability analyser

The laboratory model set up may be observed in Fig.1 which is simple and coal of the selective size is poured in the hopper and feed through vibratory feeder which ensures that the particles form monolayer of coal spread on small moving conveyor belt and passes between the detector and x-ray generator. The connected computer with data acquisition system records the data in the required format. The measurement determines particle's area, mass, and ash of the particles and it was observed that mass values are consistently accurate.

X-ray generator and detector assembly

(a) X-RAY GENERATOR

Multi-energy X-ray generator source is used which consists of an integrated X-ray tube, dual output high voltage power supply and a filament supply with control circuitry. The software configures and initializes the X-ray unit through an RS 232 port. The reference voltage, current, etc. can be set and the X-ray source enabled by issuing commands through the RS 232 port from the master controller. Once initialized and enabled, the generator emits X-rays in a continuous spectrum of energies. The X-rays are collimated into a straight beam. All reflected and scattered rays are filtered out to reduce measurement errors.

(b) DETECTOR ASSEMBLY

The electrically generated X-ray having a range of energy, it is the detector which senses energy with a single, dual and multiple level detectors. In the current study dual detector was used. The arrangement levels of detectors are shown in the Fig.2.

The detector assembly comprises two sets of sensor arrays fitted end to end. Each detector board comprises 64 dual energy detector channels at 1.5 mm pitch. 64 low energy detectors are mounted on the upper side of the board (closer to X-ray source) and 64 high energy detectors on the underside. A filter is fitted in between whose thickness depends on the level of X-ray energy which requires to be filtered out. The total width of the assembly is ~ 100 mm. The signals from the detector boards are processed by a signal processing board in the sensor assembly, and data corresponding to each pixel is converted to 16 bit format. 512 bytes (128 pixels x 2 bytes x 2) of data corresponding to the ten detector boards can be read out of the sensor assembly every 512μ sec. The data at a rate of 5Mb/second will be read from sensor assembly through a USB interface by the software at master controller (MC).

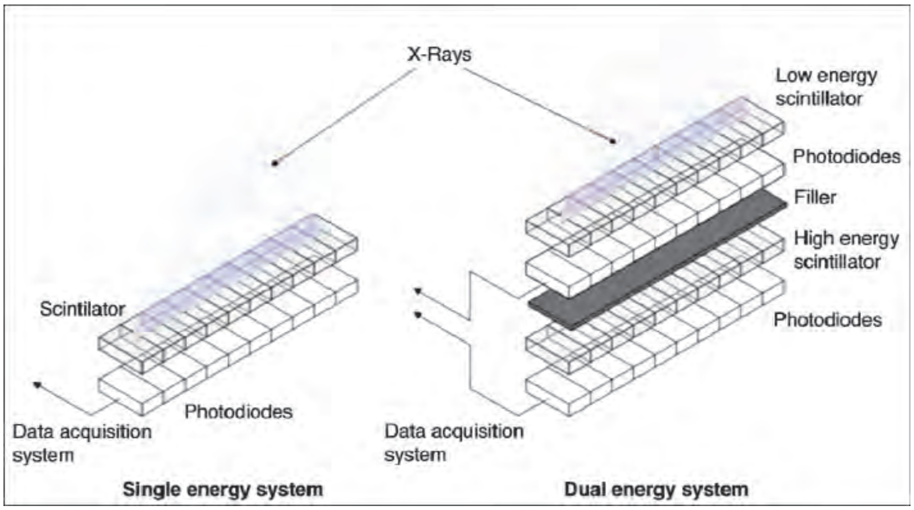

Fig.2 Detector assembly comprises signal and double sets of X-ray sensing

(c) DUAL ENERGY-X RAY TRANSMISSION SENSOR

An electrical X-ray generator applied to the broad-band X-ray radiation which falls on the moving conveyor (at a rate of 3 m/s) caring feed material to be scanned which works like a line-scan camera, record transmitted X-rays passing through the material. The formats of recorded data are in digital image data. As the name implies there is two sets of sensor system (Fig.2), each capturing the image of the material in different X-ray energy levels (low and high). Each particle attenuates the X-ray radiation received, thus decreasing the modulation amplitude of the sensor to varying degrees, so that these images areas appear in different shades of grey.

In the low/soft energy X-ray region falls from few hundred eV to 20 KeV, direct detectors image sensors are utilized, which provide high detection efficiency and high energy resolution. The hard X-ray region with energy higher than soft X-ray, having higher penetration efficiency for the object is utilized for industrial application. The Si PIN photodiode, Si APDs, and CCD area image sensor are used for soft X-ray, while scintillator detectors are wildly used for hard X-ray detection purpose.

The attenuation depends on both the thickness and atomic density of the material. Images of different atomic densities are transformed into images of different spectral ranges, which make it possible to classify different pseudo colour pixels according to specific atomic densities which regardless of material thickness.

Weight and density determination

The X-rays have the ability to penetrate matter and interact with atomic species. The material under investigation is irradiated with X-rays of known incident energy and the attenuation is accounted for coherent, incoherent scattering and absorption. This has been well described by Zou et al (2008) and applied for the determination of densities of materials which can be expressed as degree of X-ray transmission with material density and thickness.

$I = I_O x e^{-\mu \rho d}$

Where, I_O = incident radiation, I = transmitted radiation, d = absorption path length, μ = absorption coefficient and ρ = product density

Knowing the incident radiation and the constant absorption coefficient of the material and measuring the transmitted radiation using a X-ray line detect or the weight per area (ρd) may be derived.

Washability curve

The software running on the computer may run further to processes the acquired data for the determination of densities, weight, particles size and the ash, which on rearrangements can provide the results as per the requirement in tabular format and the entire four washability curves may be plotted as desired.

Results and discussion

The electrically generated X-ray having a range of electromagnetic energy, it is the detector which senses energy with a single, dual and multiple level detectors. In the current study dual detector was used (Fig.2).

It is a well-known fact that attenuation depends on both the thickness and atomic density of the material. Images of different atomic densities are transformed into images of different spectral ranges, which make it possible to classify different colour (pseudo) pixels according to specific atomic densities. This is accomplished almost regardless of the thickness of the material. Initially, a few thousands of coal samples were tested as preliminary investigations and the observations recorded with the laboratory model, is discussed here as below:

After getting the acquisition of data it was processed as primary calibration, secondary and tertiary calibration.

PRIMARY CALIBRATION

1. The preliminary investigation shows deviations in the ash%, size and weight. The ash% generated from look-up table needs application of suitable correction factor with the actual standard laboratory results as can be observed in Figs.3 and 4.
2. The variations in the results are not consistent, it varies with the size and the correction factors application need to be applied depending on size in the processing software.

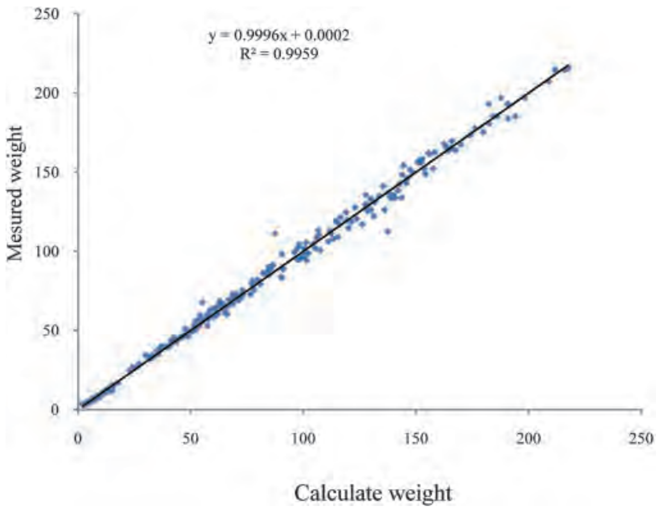

Fig.3 Showing deviations of calculated and measured weight

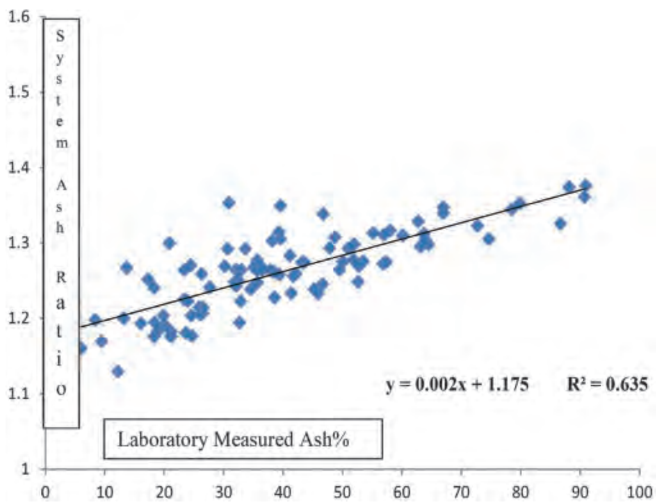

Fig.4 Measured and calculated ash percentage showing poor correlation

3. Since there is a single source of X-ray radiation it was considered that due to deviation from tangential nature the result may varies with respect to change in sample position. The samples were tested covering entire width of the conveyor and taken into consideration during finalization of the software.

Secondary and tertiary calibration

The system requires several levels of calibrations, some automatic in nature which is a part of the equipment start-up routine and the others are manually activated. Each of these needs to be done carefully at initial stage.

The first level of calibration is the automatic system calibration. In this stage, when the system is switched on, each of the cards is exposed to collimated multi-energy X-rays. Depending on sensitivity of each pixel, there is scintillation generated which is translated into a digital count. These are raw counts and are likely to vary quite drastically from pixel to pixel. When system is instructed to auto-calibrate, counts in each of the pixels are equalized through an internal gain mechanism. This is the base count from which the system starts its measurements.

In the second level of calibration, which essentially calls for some degree of manual intervention in the initialization, the system is used to measure and analyses a combination of Etalons (calibration blocks) with different heights and densities. When the second level of calibrated data is to be done, the system is given a logic for inclusion of the calibrated value through a simple "if" logic whereby if the observed value is >XXXX<, then a particular factor is to be applied this enables higher level of accuracy for the size range.

Third level of re-calibration is to compensate for X-ray hardening. X-ray hardening happens when mass per surface area exceeds a critical level. After this level, the attenuation curve becomes flatter due to which there can be an erroneous reading of data at higher densities and higher heights of the particles and it will have to be compensated.

Correction for X-ray hardening

Both low energy and high energy show a tendency towards reduced attenuation as the particle gets bigger and/or higher density. Typically, for high energy this takes place around 17000 counts and for low energy it is 12000 counts. The hardening effect of X-rays causes the downward sloping curve to become flatter which in effect minimizes the mass per surface area of the particles beyond the threshold values as above causing distortions in estimation of particle weight and ash as well. The correction is induced in the form of eliminating the lowest height readings to re-configure the values for high and low energy readings below 17000 and 12000 respectively.

Comparative analysis of laboratory float and sink test with system generated data

Raw coal was crushed to -75 mm and screened at - 13 mm was fed to the input belt in the analyser. The samples were manually loaded on the hopper from where an SS troughed electromagnetic feeder (EMF) which is having variable settings for amplitude and frequency control puts the material onto the conveyor belt. An appropriate frequency was chosen so that particles are fed to the belt below discharge point of the feeder to form a monolayer and they do not overlap with each other. The material is dropped onto belt where it is subjected to a particle by particle analysis using dual energy X-ray transmission system to determine size, weight and ash% of individual particles. Once the data is captured by the analyser through the data acquisition system, it is stored in the computer with a date and time tag. This file is then retrieved by the system and subjected to two rounds of data filtration. First stage filtration is to remove the noise factor that creeps into the data due to radiation dispersion, reflection and hazing caused by changing particle shape and composition. Second stage filtration aims to eliminate the extremes in observed values based on running a trial equation of the behaviour of the system in relation to particle-wise analyses vis-à-vis laboratory analysis.

The system has some stray readings which tend to disturb and distort predictive capacity of X-ray based coal washability analyser system. The stray readings are caused by a whole host of factors including power fluctuations, radiation hardening due to sudden changes in material size and composition, particle instability on the belt, vibrations induced in the system due to ambient conditions and several other factors. It needs to be eliminated by a standardization process which is not arbitrary but based on the standardization process of imparting a corrected and linearized regression relationship between high energy particle-wise composite data and weight of the particle. Once this is arrived at, the outlier observations that are off the predicted line by a significant percentage, typically ± 20%, are eliminated from samples being evaluated. Then the corresponding low energy weighted averages are calculated

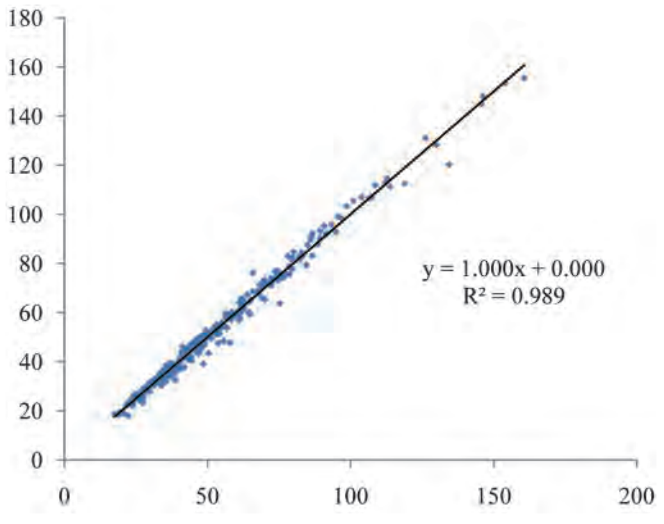

Fig.5 Lab model physical data vs online module data

for each particle to determine the ash% in the partially corrected raw data. Here again, the filter is applied and those which are off the predicted curve values beyond ± 20% are eliminated from the analysis. The edited values are then correlated with the lab data in terms of relevant weight and ash% for each category.

Once both the results were over, the data is being correlated with each other by a category migration method. The traditional washability method, float and sink test is being conducted using specific gravity to get yield and ash% for each sample. The specific gravity in X-ray based analyser system use as a proxy for ash%, as scientific wisdom dictates that ash% in coal particles increases when subjected to float and sink tests at higher and higher specific gravities.

The two sets of data have to be equated in terms of a proxy factor that can be considered a close clone of each other. We have taken the float specific gravity of 1.26 to be the base value of coal with zero ash% and moving up by 0.01 in terms of specific gravity for every unit increase in ash%. Since the first stage of ash% in float and sink analysis is normally < 1.4 specific gravity, this was translated into ash% level at around 12%. The remaining categories were then suitably adjusted to give one common base for comparing the two sets of results.

Since, it was handling limited sizes, viz., 13 - 25, 25 - 50 and 50-75 mm, the software classifies each of the particle sizes into its respective categories and does the averaging for that category in terms of both ash% and weight. The data is then processed to cumulate the weight and weighted ash% and the table generated on that basis. Then based on all the observations and correction made to the software the output of the analyser are presented in the Table 1 for the size analysis and washability graphs for the respective size below:

TABLE 1: SIZE ANALYSIS OF COAL, PASSING AFTER X-RAY BASED COAL WASHABILITY ANALYSER

Size	Wt (kg)	Wt%
50-75	9.5	10.61
25-50	60.0	67.04
13-25	18.0	20.11
-13	2.0	2.24
	89.5	100.00

The coal passed through the analyser and after acquisitions data was screened for size analysis. Each and every size was subjected to float and sink analysis. The weight and ash were determined followed as per standard and washability table and curve were plotted. The washability curves (Figs.6, 8 and 10) are constructed based on the laboratory float and sink data for the coal size fraction 50-75, 25- 50 and 13-25 mm respectively, which is done in the ideal condition using desired chemicals flowed as per standard.

The washability curves (Figs.7, 9 and 11) were

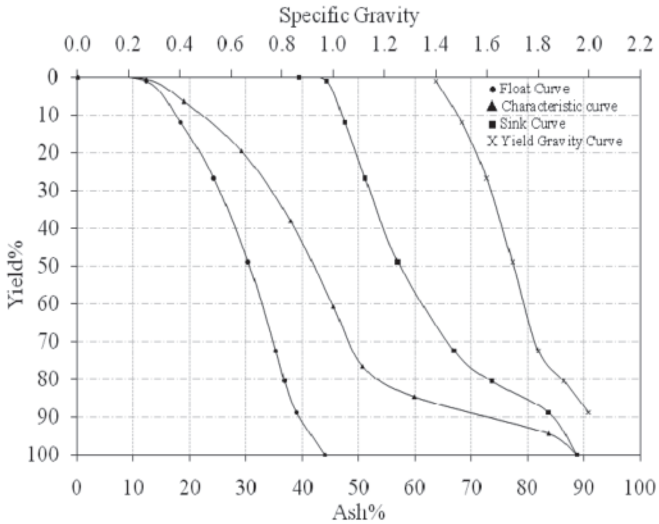

Fig.6 Standard laboratory F-S data for size 75-50 mm

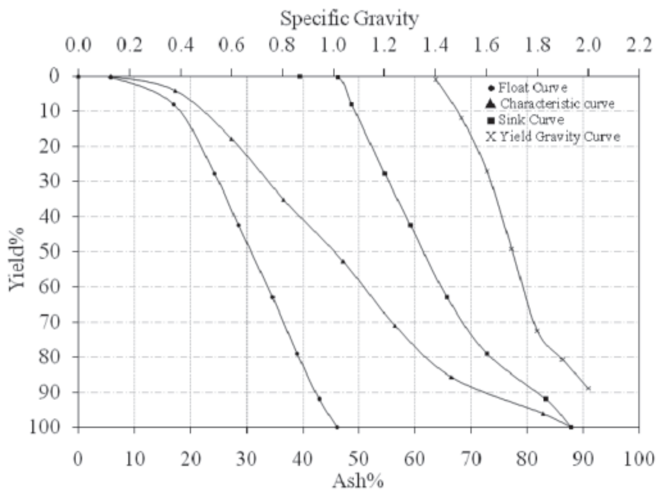

Fig.7 X-ray based (F-S) analyzer data after processing, size 75-50 mm

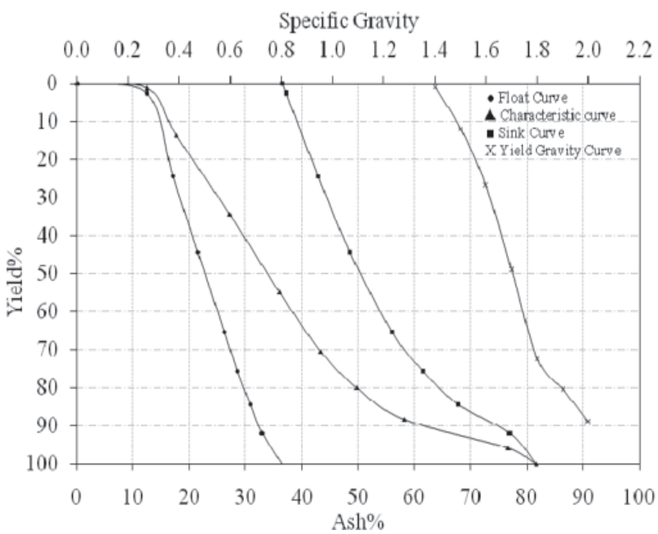

Fig.8 Standard laboratory F-S data for size 50-25 mm

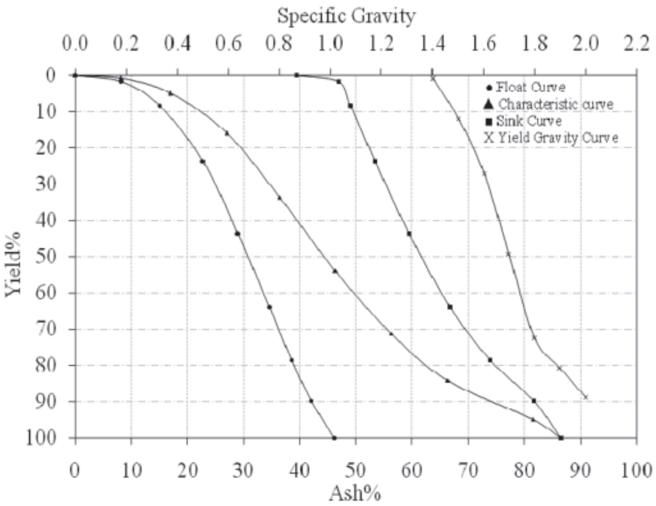

Fig.9 X-ray based (F-S) analyzer data after processing, size 50-25 mm

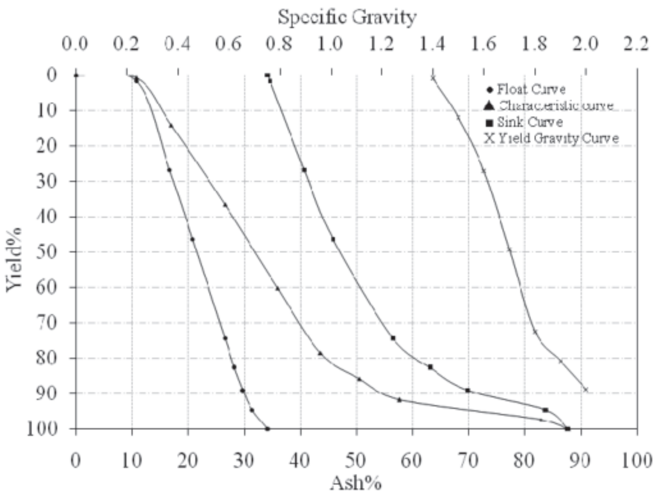

Fig.10 Standard laboratory F-S data for size 25-13 mm

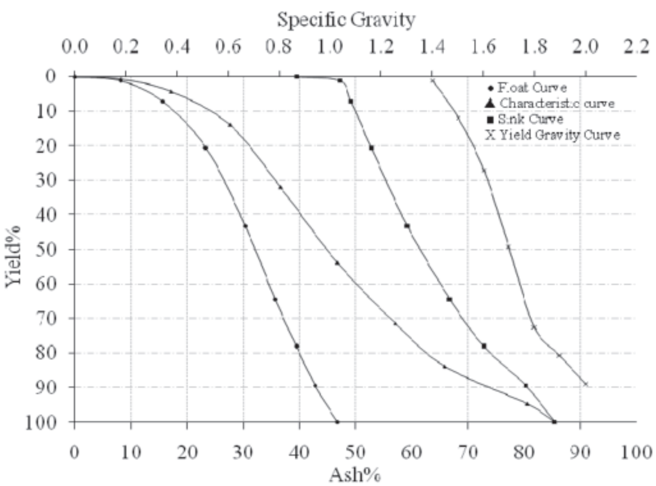

Fig.11 X-ray based (F-S) analyzer data after processing, size 25-13 mm

constructed based on the X-ray based analyser data acquisitions system after application of data filtering and processing for the size fraction 50-75, 25-50, 13-25 mm respectively. The comparing of data with respect to their size can be observed overall ash for the larger size (50-75 mm) data and float and sink results in good agreement with the laboratory standards float and sink. While going to relative smaller fraction this deviated larger from the reality, which is higher and provides side of the online data. The works need further improvement to narrow down the gaps of washability analyser results to be in the acceptable limit.

Conclusions

The X-ray based coal washability analyser is not using any hazardous chemical in processing to get the results almost in real time data analysis. This results in improvement of the performance of coal preparation plant for maximum possible recovery which may be obtained during the process of coal washing.

The X-ray based coal washability system provides more details information than the standard laboratory float and sink method. The data is having each and every particles detail like size, density and ash which on further processing one can set the classlimits as per the desired requirements. The fact that the results are stored in a data base offers the possibility to reevaluate entire data sets under different aspects which cannot be done with the lab results.

The analyser is X-ray based and it has the capability which may handle up to atonic level of analysis and the advantage may open up the opportunities for in-depth analysis of particle of interest for their compositions and associated physical and chemical properties. This may entirely change the processing technology at the user point of view.

The limitation of time and accuracy of analysis is particle size with the speed of analysis, to lower the particle size much higher time is required for better resolution of detectors requiring lead to higher analyzing time to make the process slower.

It may be observed that overall ash of the larger size (50-75 mm) data and float and sink results in good agreement with the laboratory standards float and sink. While going to relative smaller fraction it deviated larger from the reality, which is providing higher side of the online data. Further studies are being carried out to narrow down the gaps of washability analyser results, to be in the acceptable limit.

Acknowledgements

This work is funded by Ministry of Coal, Government of India. Authors are thankful to Director, CSIR-CIMFR, Dhanbad for his kind permission to publish the paper. Authors are also thankful to coal preparation and carbonization research group for their cooperation.

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