Introductory to the Application of Portable X-Ray Fluorescent Spectroscopy (PXRF) on the Investigation of On-site Ash Content Analysis

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Abstract—**Ash content of coal refers to the non-combustible residue left after the combustion. It is mostly expressed as a percentage of the original weight which can be used as the indicator for the quality of coal. By the circumstances in material classification method, the conservative standard analysis becomes one of the time and energy consuming process in coal quality determination. Since the laboratory-based analyses can be a bottleneck for further coal utilization, the development of on-site analysis procedure can be one of the alternative for overwhelming the limitations. Currently, a Portable X-ray Fluorescence spectroscopy (PXRF) is generally applied as the main instrument in on-site investigation for materials' characteristic. It offers an in-situ analysis which can precisely identify some of the compositions in coal, so that the limitations and difficulties in ash content determination can be diminished. In this study, the PXRF is applied for the analyzing of the composition in 24 sub- bituminous coal samples from the northern part of Lao PDR. Under the linear multiple regression analysis for estimating the relationship between mineral compositions in coal samples and the total ash content in the sample were conducted to determine the linear relationship formula. The result of the study exhibits the possibility in applying this formula in the instantaneous-estimation of total ash content in the coal sample.**

Index Terms—**coal, Ash, PXRF, regression, multicollinearity**

I. INTRODUCTION

Coal ash is one of the components in coal that can be collected after the combustion in form of the residue from the combustion. The chemical composition of coal ash sturdily depends on geological and geographic factors related to the coal deposits, method and condition of combustion and the precipitation technique [1]. The major composition of coal ash is qualitatively based on its parental materials such as soils and shales which can designate the characteristic of ash.

Oxidized compounds of Si, Al, Fe, and Ca can be accounted for nearly 90% of the composition of ash. Other elements, such as Mg, K, Na, Ti, and S occur as minor constituents with a small percentage of the bulk composition. Generally, all other elements occur in the parts per million range and, collectively, seldom exceed 1% of the bulk composition [2].

The analysis of ashes is complicacy since their composition is highly inconstant, extremely heterogeneous and most of them are classified as silt which are very fine particle size. Various techniques have been applied to influence the characteristics of ash to optimize its utilization, increase its value and minimize the disposal cost [3]. These qualitative characteristics of coal ash are determined by the composition of coal and combustion parameters. So, it is imperative to quantify the elemental composition of coal ash.

There are various analytical techniques capable for characterize coal ash with their own advantages. Among simultaneous multi-elements analysis techniques, X-ray Fluorescence (XRF) instrumentation performs lower capital cost and is cheaper to use than Neutron Activation Analysis (NAA), is faster than ICP-AES. XRF techniques require less sample preparation time, cost and avoid the use of hazardous analytic chemicals.

Recently, X-ray fluorescence analysis has been developed into portable analytical device and emerged as a very powerful technique for in-situ elemental analysis of environmental samples [4].

The Portable X-ray Fluorescence spectroscopy (PXRF) offers non-destructive analysis of both quality and quantity of the mineral composition in multi-element samples. This instrument provides a competence in conducting a reliable on filed-chemical analysis with a short period of time.

The application of PXRF in the environmental and mining sector to determine elemental distributions and quantifying element contents were found to be reasonable. It can generate reliable, high quality elemental concentration data for metal-contaminated soils [5].

[6] mentioned that the PXRF was validated PXRF as a suitable alternative to ICP-AES for estimating the percentage of elements in the measurement of metal-contaminated soils but it requires appropriate sample handling, preparation, and instrument calibration, in order to generate the data which are equivalent or better than ICP-AES.

PXRF has been applied and stated to be one of the cost and time effective for many aspects such as onsite geological surveying and environmental monitoring.

Reference [7] applied the PXRF for analysing pedogenized sediments of a basin in Italy. The results demonstrated that the instrument can rapidly provide data for some significant components which can reduce cost and time for the analysing of explicating the pedogenic processes in the basin area.

Lauren L. *et al.* [8] studied the mineralisation of nickel by using PXRF as the main instrument. The results demonstrate the high capability of PXRF in the support of delivering a robust dataset to help differentiate the area that contains zones within a komatiite flow. The instrument also enhanced the advantages in handling large numbers of samples with a short period of time with high precision in the determining the different aspects and interests in lithogeochemistry.

[9] suggested that PXRF has a capability to become a routine part of the characterization of geothermal cuttings during geothermal exploration and well drilling. By comparing the analysis results from untreated geothermal drill cuttings with laboratory XRF and PXRF. The comparison demonstrated that the portable XRF results were accurate when defining some of elements, particularly for those with atomic numbers greater than 17.304. As the chemical results are accurate, rapid and inexpensive so that the application for onsite PXRF can be used to describe lithological boundaries and potentially correlate between drill holes, therefore improving geologic, stratigraphic and hydrothermal alteration models of the geothermal field.

In addition, the PXRF can be applied as a useful device in association with the multiple linear regression analysis for onsite investigation between chemical compounds within the study area. [10] studied on the possibility in predicting soil properties based on PXRF data. The study's result indicated that the variables obtained by PXRF also allowed the spatial prediction of soil properties related to soil fertility with high accuracy in the estimation and can be lead to the generation of accurate spatial map of variations at low cost and without generating residues.

Figure 1. Portable X-ray Fluorescence spectroscopy (PXRF) which is widely applied as on-field material characterizing. It is capable for the determination of elements in form of parts per million or percentage of elements. However, the determination some light elements cannot be conducted.

With this capability in conducting instantaneous in-situ chemical analysis in association with statistical study, the coal ash content analysis can be enhanced. In this study, the correlation between the value of ash content and the concentration of significant mineral compositions in the ashes, defined by PXRF, were analysed, using multiple regression analysis to determine their relationship. Fig. 1 demonstrates the PXRF applied in this study.

II. METHODOLOGY

The sub- bituminous coal samples from the Northern of Laos PDR were collected. Theses samples were prepared and analyzed under the ASTM D 3174 – 12 [11] and ASTM D 5865 – 04 [8] for ash composition analysis and gross calorific value of coal respectively. Both accomplished analytical results were analyzed under the linear analysis to determine the mineral that relates to the existence of ash after the combustion. As a final stage, the multiple regression analysis to determine the relationship between significant mineral compositions in ash and the total mass of the ash. As stated earlier, twenty-four coal samples, determined to be sub-bituminous class, from the northern part of Lao PDR were used as the samples in this study. The size of all coal samples was grinded and classified to be under 80 mesh for further analysis.

According to [12], the dried coal samples were installed into the cover and gradually heated to reach 450 to 500 ℃ for one hour. Heated samples' temperature was raised to 900 ℃ by the end of the second hour. Remove the containers from the furnace cool under conditions to minimize moisture pickup, and weigh. The residual, left after the heating process, then allowed to cool then weighted.

The ashes samples were prepared following ASTM D5016 - 08e1 [13]. The process began with placing a weighed analysis sample of coal in a cold muffle furnace. They were heated gradually until the temperature reaches 750 ℃ and the samples reached a constant weight. The ashes, retrieved from the process, were then cooled, ground to pass a 74 μm standard sieve, and reignited at 750 ℃ for 1 hour. Materials such as residual ashes, fly ash, or bottom ash must be ignited until a constant weight is reached at 750 ℃ and cooled in place.

Since the standard method for PXRF has not been release, the analysis on ashes composition were conducted following ASTM D 4326 – 04 [14] at the sample preparation state. For the analytical apparatus, the ashes sample were place in a holder, provide by the manufacturer.

The samples were analyzed for 90 seconds, as stated by the manufacturer's instruction, to collect all the signals from both Light Elements (LE) and significant ash composition. The analysis data were collected as the required data input for linear multiple regression analysis.

To study the relationship between the ash content (independent variable) and major elements in coal, the results from ash content analysis were analyzed, using the correlation analysis and stepwise multiple linear regression.

The correlation analysis was assigned in order to investigate the existence of multicollinearity between the significant compositions of coal samples, while the stepwise multiple linear regression was conducted to explore the relationship between independent variables (ash composite) and the dependent variables (ash content).

These analyses were conducted with the intention to decide the selection of the independent variables in the relationship equation. The multiple linear regression equation applied in this study is established in (1).

$$Y = a + b_1X_1 + b_2X_2 + \cdots b_nX_n \qquad (1)$$

where Y refers to the ash content in coal samples. Xn is any elements, detected in coal by the PXRF (%). "a" is stated state the Y – intercept while bn are the regression coefficients retrieved after the multiple linear regression analysis.

III. Results & Discussion

A. Ash Composition Determination Results

The coal samples were prepared and analyzed according to the selected ASTM method. Consistent with the capability of the PXRF, the determination of carbon and volatile matters in coal samples were reported as Light Element content (LE). Significant composition in ashes sample Silica (Si), Iron (Fe), Calcium (Ca),

Aluminum (Al), and Sulphur (S) were selected as the significant composition of ash. Table I. exhibits the PXRF's results, indicating that the ash composition from coal samples applied in this study, were fluctuated. The ash content in the samples were detected to be ranged from 9.44 to 61 percent by weight of coal. LE were determined to be the component that were mostly found in all of ash samples ranged from 72.66 to 86.12 by weight. However, for other mineral compositions, the instability in the analysis' results were sensed due to the variation of the samples.

B. Ash Composition Determination Results

Table II demonstrates the correlation analysis results of the significant element in the samples. Conforming to the results, the multicollinearity between the significant compositions were detected. This is a problem that occurs when high correlation of at least one independent variable with a combination of the other independent variables. In this analysis, Aluminum and Silicon demonstrates the correlation between each other at 0.804. The high correlation can cause the effect to the error in the multiple regression analysis in form of a controversial value of the regression coefficient in the regression model. Since the multicollinearity of the samples was perceived, the stepwise multiple regression analysis was applied as the main analysis to neglect the effect of the multicollinearity between Al and Si.

TABLE I.   Ash and Chemical Content of the Coal Samples

Sample	Ash Content (%)	LE (%)	Si (%)	Fe (%)	Ca (%)	Al (%)	S (%)	Sample	Ash Content (%)	LE (%)	Si (%)	Fe (%)	Ca (%)	Al (%)	S (%)
C 01	16.03	81.33	4.64	4.62	3.49	2.29	2.07	C 13	23.58	80.22	7.52	4.25	0.90	4.07	0.75
C 02	12.88	83.85	3.82	4.10	3.38	1.85	1.72	C 14	44.54	76.00	9.62	4.76	1.37	4.54	1.49
C 03	11.93	84.82	3.07	4.25	2.99	1.54	1.54	C 15	39.04	74.71	9.63	4.87	1.51	4.59	1.77
C 04	9.44	85.72	2.14	4.22	3.99	1.15	1.89	C 16	22.37	76.25	3.62	1.10	13.29	1.13	0.45
C 05	10.17	84.84	2.69	4.63	3.01	1.40	2.39	C 17	15.66	81.57	4.51	4.59	3.48	2.31	2.04
C 06	23.20	82.82	6.43	3.78	0.74	3.43	0.65	C 18	10.99	84.78	3.04	4.44	3.21	1.68	1.75
C 07	44.44	74.33	10.04	4.65	1.43	4.81	1.60	C 19	9.77	86.12	2.38	3.75	3.65	1.59	1.73
C 08	39.31	76.43	9.33	4.50	1.43	4.63	1.62	C 20	9.54	84.23	2.59	4.71	3.26	1.57	2.65
C 09	23.58	81.19	6.55	4.52	1.01	3.29	1.94	C 21	38.44	77.63	12.26	4.91	0.73	1.92	0.62
C 10	39.59	76.90	8.89	4.84	0.86	4.49	1.96	C 22	61.45	72.66	11.09	5.46	2.06	5.28	0.37
C 11	33.04	78.59	8.00	4.37	0.82	4.02	1.73	C 23	42.38	76.60	11.67	4.48	0.57	3.41	0.69
C 12	9.62	83.75	3.53	4.55	3.29	1.89	1.80	C 24	11.47	80.51	3.77	2.12	8.49	1.71	0.95

TABLE II.   The Correlation Analysis Results

		Ash	LE	Fe	Ca	S	Al	Si
	Ash	1.000	-.920	.371	-.410	-.461	.856	.936
	LE	-.920	1.000	-.106	.116	.480	-.770	-.852
Pearson Correlation	Fe	.371	-.106	1.000	-.865	.353	.457	.419
	Ca	-.410	.116	-.865	1.000	-.166	-.577	-.544
	S	-.461	.480	.353	-.166	1.000	-.222	-.470
	Al	.856	-.770	.457	-.577	-.222	1.000	.804
	Si	.936	-.852	.419	-.544	-.470	.804	1.000

Table III exhibits the results of the analysis. Since the multicollinearity of the samples existed, the stepwise multiple regression analysis was applied as the main analysis to neglect the effect of the correlation between Al and Si. Table III exhibits the results of the analysis. The results from the analysis designated three model that can describe the relationship between significant coal composition and the ash content.

TABLE III.   THE RESULTS OF MULTIPLE LINEAR REGRESSION IN THIS STUDY

Significant composite	Model 1	Model 2	Model 3
Constant	-1.575	142.877	178.874
LE	-	-	-2.184
Fe	-	-	2.562
Ca	-	-	-
S	-	-	-
Al	-	-	-
Si	4.245	2.512	1.681
R Square	.876	.931	.945
Adjusted R Square	.871	.925	.937
Std. Error of the Estimate	5.461	4.164	3.809
F	155.646	142.291	115.072

"-" = Excluded by statistically insignificant (P<0.0001)

The comparison between these three models demonstrates that the third model, accompanying SE, Si and Fe as the model input were the most appropriate model in defining this relationship with the standard error of estimation at 3.809 and F variance equal to 115.072%. This statistical parameter demonstrates that the relationship between three compositions can be used as the predictors for the onsite ash content analysis of these samples.

Additionally, the results from the stepwise multiple linear regression analysis indicates that Ca, S and Al are not statistically significant to the relationship of the quantity of the ash content in the coal samples so that they were excluded from the analysis during the stepwise calculation process. The regression equation from the third model is expressed in (2).

$$Y=178.874-2.184(\%LE) +2.56(\%Fe) +1.681(\%Si) \quad (2)$$

In consistence with the regression equation, %LE, %Fe and %Si refer to the content of each components, obtained from the onsite quantitative analysis, using PXRF. This can be implied that LE, Fe and Si are sensitive to the change in ash content of coal samples. Si and Fe were the elements that can increase the portion of ash in coal, while LE behaves in the opposite direction due to their volatilities.

C. Model Evaluation

Table IV demonstrates the evaluation results of the regression model from (2). The results exhibit the error range from 0.55 to 56.61% when apply the linear relationship for the estimation. Even the statistical indicators designate the high compatibility in using the retrieved equation in the estimation, but high errors in the application of this linear equation cannot entirely represent every samples in the study.

TABLE IV.   EVALUATION OF THE LINEAR RELATIONSHIP FROM MULTIPLE LINEAR REGRESSION ANALYSIS

Sample	Analyzed ash content (%)	Estimated ash content (%)	Error (%)
C01	16.03	20.58	28.43
C02	12.88	12.42	3.57
C03	11.93	9.47	20.56
C04	9.44	5.93	37.24
C05	10.17	9.79	3.71
C06	23.20	18.08	22.08
C07	44.44	44.69	0.55
C08	39.31	38.57	1.88
C09	23.58	23.72	0.60
C10	39.59	37.70	4.78
C11	33.04	31.37	5.07
C12	9.62	13.32	38.47
C13	23.58	26.72	13.33
C14	44.54	40.64	8.74
C15	39.04	43.76	12.08
C16	22.37	21.02	6.03
C17	15.66	19.77	26.25
C18	10.99	10.00	9.00
C19	9.77	4.24	56.61
C20	9.54	11.16	16.97
C21	38.44	41.74	8.58
C22	61.45	52.11	15.20
C23	42.38	41.93	1.06
C24	11.47	14.57	27.01

D. Discussion

In keeping with the multiple linear regression's result, the relationship between LE, Si and Fe can use as the input parameter in the estimation of onsite ash content analysis of coal while the content of Ca, Al and S were excluded from the model due to their statistical insignificance. However, the model evaluation indicates some of errors in the estimation, thus some of the evaluation's result still demonstrate the possibility in developing this procedure for onsite investigation of ash content.

Since the variation of coal's compositions is uncontrollable, the estimation of ash content using only statistical analysis based on the limited samples can cause the error to the estimation's results.

As can be detected from the model evaluation, the regression's equation applied in the analysis cannot effectively describe the relationship among the compositions, resulting in the high range of error in the estimation.

Additionally, the fluctuation in the content of the compositions, especially the content of lighter significant elements such as Si, is one of the cause of errors in the estimation. These fluctuation is affected by the limitation of the PXF when quantifying lights element. Since PXRF requires longer time for detecting the light elements so that some of the interferences can alter the analysis' results.

Homogeneous of coal samples is another factor that amplify the error in the estimation. PXRF, capable for determining elements from the sample's surface, can detect only the composition within the pointing area of the device with inadequate radius of detector. This makes the analysis' result been not able to represent the complexity of the composition, resulting in discrepancy of the content of analysed elements.

PXRF demonstrates its capability in being applied as the main analytical instrument for onsite-ash content investigation. Nevertheless, the estimation procedure still based on the relationship, obtained from the multiple regression analysis, is based on the statistic between the group of these specific samples so that the relationship of the common significant elements of coal and the ash for the communal mine site still indistinct. In order to develop the better equation for estimating this

relationship, the numbers of coal samples from various sites of coal must be applied.

IV. CONCLUSION AND SUGGESTION

A Portable X-ray Fluorescence spectroscopy (PXRF) is applied to analyzed twenty-four sub-bituminous coal samples from the coal mine in the northern of Lao PDR. Under multiple linear regression analysis, the sensitive significant elements which are Si, Fe and LE were demonstrate their capability in being applied as the input for linear formula for on-site estimation of ash content from coal. However, the evaluation of the equation, retrieved from the analysis still cannot be applied to the other communal coal in every site.

Since the PXRF has a time limitation for the determination of chemical contents for the elements with low atomic number, so the appropriate sample handling preparation must be conducted. Since the detection time of the light element by this device can allow the environmental condition such as humidity interfere the accuracy of the determination, providing some cover during the analysis will enhance the reliability of the content of the significant elements in the analysis.

Homogenization of the coal samples is the corresponding factor that control the accuracy of the estimation. Preparing the sample using standard practice can be one of the procedure that can diminish the error from the variation of coal composition and elements.

As stated before that even the stepwise multiple linear regression is an effective tool, thus the method still not cover all the effect from the significant elemental composite to the ash content of coal samples. In order to improve the regression equation, the multiple regression in form of other relationship such as exponential relationship between the parameter should be studied.

Since the PXRF has been considered to be the onsite tool that can handle the larger number of samples, increasing in the number of coal samples will improve the compatibility of the regression model, resulting in the higher accuracy of the estimation.

Nevertheless, the classification of coal samples must be severely monitored. Coal, differs in parental material, should be characterized in term of quality so that the development of appropriate regression model that can describe the relationship between significant composition and the ash content can be accomplished.

As a conclusion, this study designates the application of PXRF in being assigned as the main tool in on-site estimation of coal properties. The results indicates the possibility in developing of regression equation for describing the relationship between significant coal composition and the ash content instantaneously without the destruction of coal samples, thus the improvement for better model for estimation should be furthered studied.

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