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PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBON DERIVED FROM RICE HUSK BIOMASS

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Abstract

Rice husk was washed with distilled water to remove the contaminants present in the rice husk. The wet rice husk was dried at room temperature. The most important properties that provide information about the fuel were proximate such as moisture, ash content, volatile and fixed carbon contents. The fixed carbon content of rice husk biochar (RHB) was measured to be 10.3 mf wt%. The experiment was carried out to study the characterization of the rice husk ash. The rice husk biomass was heated at 300˚ C for 1h in muffle furnace and grounded into the uniform powder. Then, the carbonized material obtained was socked in 1M KOH, in 1:1 ratio for 24 h and followed physical activation at 300°C for 2h in muffle furnace. The structure characterization of activated carbon rice husk biochar (AC-RHB) was identified by X-ray diffraction (XRD) technique. The carbon C was observed by Energy Dispersive X-ray spectroscopy (EDXRF). The porous nature of AC-RHB was studied by scanning electron microscopy (SEM). The carbon C, O, and Si were observed by Energy Dispersive X-ray spectroscopy (EDX). The Fourier transform infrared spectroscopy (FTIR) analysis showed the presence of a variety of functional groups of AC-RHB.

Keywords – Rice husk biomass, Activated Carbon, XRD, EDXRF, SEM-EDX, FTIR.

Introduction

The biomass can be considered as the most easily available and useful bio-material in the current days. Because of the renewable nature, the biomass is widely used for various purposes such as direct source of heat generation, as the feedstock for thermochemical and biological conversion to produce useful fuels or chemicals. [Monoj Bardalai. et al (2016)] Biomass materials agricultural from residues such as straw, bagasse and groundnut shell, coffee husk and rice husk as well as residues from forest-related activities such as wood chips, sawdust and bark having high energy potential. [McKendriya P

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(2002), Bidayatul Armynah, et al (2018)] These biomass wastes are one of the main assets for renewable energy. Consequently, there are numerous prominent technologies to transform biomass into energy. [Lim. J.S., et al (2012), A. Anchan Paethanom, et al (2012)] In general agro residues normally have the following compositions on moisture and ash free basis: 50% carbon, 6% hydrogen and 44% oxygen. The moisture content varies over wide range from oven dry to about 90% on wet basis and ash content varies from 0.5 to 22%. [Zhao. R., et al (2015), Purakayastha, T.J., et a; (2016)] The term activated carbon is basically referred as carbonaceous materials [Cuhadaroglu D, et al (2008)], with high porosity [Hayashi J, et al. (2000), Yacob AR, et al. (2008) 2-6], high physicochemical stability [Zhu Z, et al (2008)7-1]. Activated carbon is also called as activated charcoal or activated coal [Hiremath MN, et al (2012)] and sometime called as solid sponge. [Zanzi R, et al. (2001)] Activated carbon usually prepared from organic matter that contain of highly carbon, so agricultural waste was interesting choice because of its low cost and it was a source of renewable energy. [A. Buasri, et al (2013), J.C. Moreno-Pirajan, et al (2010), Y. Sudaryanto, et al (2006)] In the presence investigation, rice husk biomass was characterized for proximate analysis viz. Moisture(M), Ash (A), Volatile Matter (VM) and Fixed Carbon (FC). The activated carbon was prepared from rice husk biochar and this AC-biochar was characterized by XRD. Energy Dispersive X-ray Fluoresces (EDXRF) was employed to examine the elemental concentration of biochar. The rice husk biochar samples were also analyzed by Scanning Electron Microscope (SEM) equipped with an energy dispersion X-ray Spectroscopy (EDX) and FTIR for identification of pore size and functional groups.

Experimental

Preparation of Activated Carbon

Rice husk washed with distilled water to remove the impurities such as dust then the wet rice husk was dried at room temperature. Rice husk sample were weighed and then placed into furnace, temperature at 300˚ C for 1 h, and the sample were weighed. The rice husk ash sample was grounded and sieved to the average particle size. Then, 5.6 g of KOH was added to 100 mL of distilled water. The mixture of KOH solution was then stirred. After that, the rice husk sample were soaked in this KOH solution for 24 h. Then the impregnated activated carbon is heated at 300˚ C for 2 h.

Characterization Methods

The rice husk biomass (RHB) was subjected to carbonization at 300˚ C for 1 h. The rice husk biomass and biochar samples were characterized for proximate (Moisture, Volatile Matter, Ash Content and Fixed Carbon). Moisture Content was determined according to MOC63U Moisture Analyzer. Moisture content was also obtained by the sample was placed in the oven at 105˚ C for 2h. Then the sample was cooled in desiccator and reweighed again. The moisture was less than 10 mf wt%. For volatile matter sample was heated at 900˚ C for 30 min; this mass loss is attributed to volatile matter. For ash content the sample was heated at 700˚ C for a minimum 5h. The difference between the initial and final weight of the sample represents the ash content. The weight of the original sample, subtracted byits moisture content, ash content and volatile matter content (as determined by the aforementioned proximate analysis) corresponds to the stable carbon fraction of that sample and hence, his fraction is termed ‘fixed carbon or fixed-C fraction’. Figure 1. shows the schematic diagram for preparation of activated carbon from rice husk biomass.

$$\text{Moisture Content} = \frac{\text{initial mass} - \text{moisture mass}}{\text{initial mass}} \times 100 \qquad (1)$$

$$\text{Volatile Matter} = \frac{\text{moisture mass} - \text{Volatile mass}}{\text{initial mass}} \times 100 \qquad (2)$$

$$\text{Ash Content} = \frac{\text{Ash Mass}}{\text{Initial mass}} \times 100 \qquad (3)$$

$$\text{Fixed Carbon Content} = 100 - [\text{Moisture} + \text{Volatile} + \text{Ash}] \qquad (4)$$

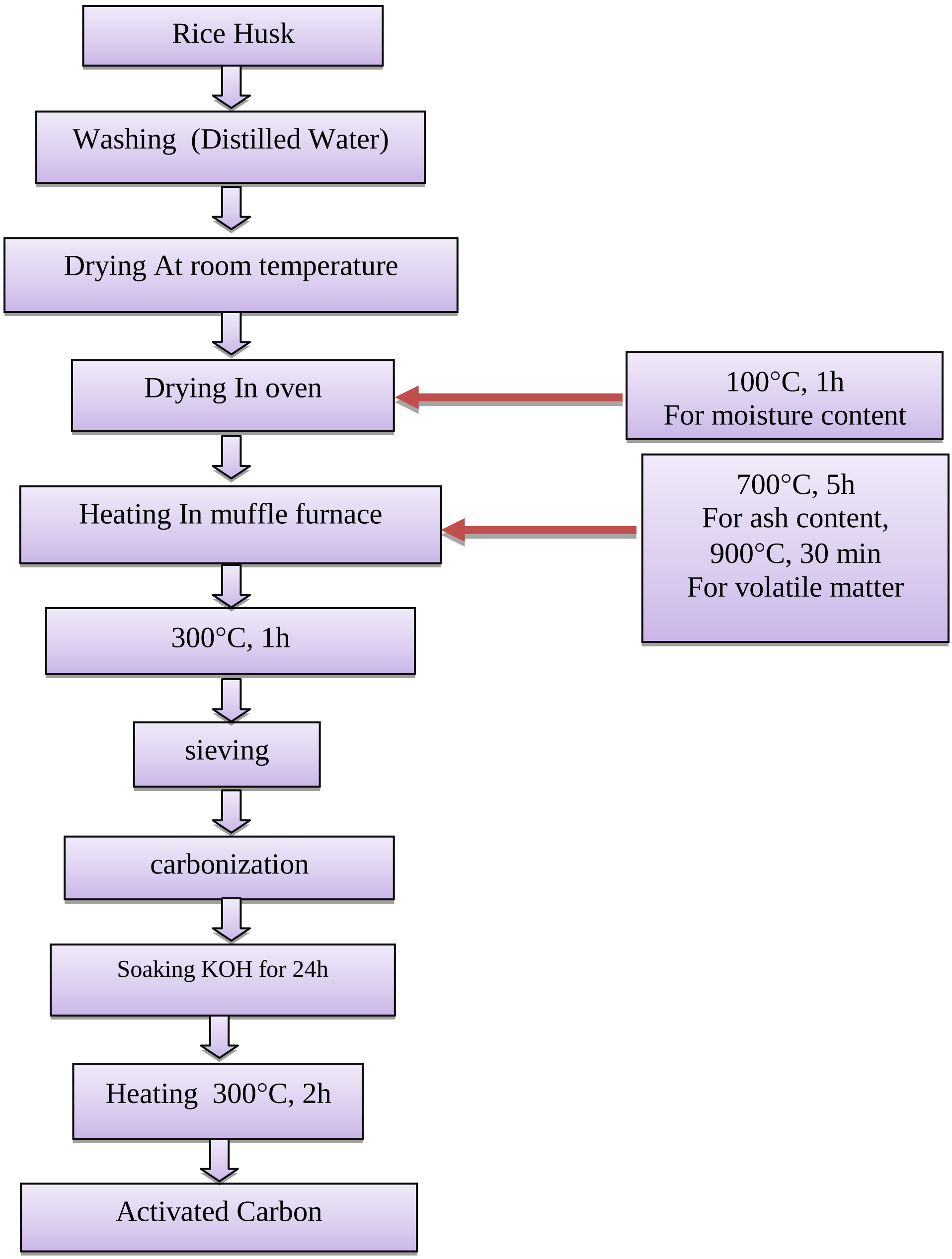


Figure 1: Schematic diagram for the preparation of activated carbon (AC) from rice husk biomass

Results and discussion

Proximate Analysis

The results of the proximate analysis of rice husk are also presented in moisture-free weight percentage, mf wt %. Table.1 shows the properties of the raw rice husk. From proximate analysis, it is observed that rice husk contains its moisture was less than 10 mf wt %, a large percentage of volatile matter, 72.7 mf wt %, a moderate percentage of ash content, 17 mf wt %, and a small percentage of fixed carbon content 10.3 mf wt %. Generally, a raw biomass has high volatile matter content where 80 - 90 % of biomass is combusted in the form of volatiles. Since most of the energy is stored in the volatiles, volatile matters are highly reactive, which makes the combustion process more difficult to be controlled. [Smith, J.L., et al (2010)] Hence, low content of volatile matter is desirable because it is an indication that the combustion process is easier to be controlled. The result suggested that the fixed carbon content was affected by the higher ash content of RHB, which inhibited the formation of aromatic carbon during the thermochemical conversion process. [Angin, D., et al (2014)] It is a common consensus that high ash content is likely to cause fouling at the current collector in DCFC. In addition, a fixed carbon is one of the important elements that determine the quality of biomass as a fuel cell. From these results were neither bad nor good. Hence, further study it requires to reduce the volatile matter and to more produce fixed carbon.

Table 1: Characterization of rice husk biochar

Proximate analysis result of rice husk biochar (wt% dry basis)			
Moisture content	**Volatile matter content**	**Ash content**	**Fixed Carbon Content**
9.54	72.7	17	10.3

X-ray Diffraction (XRD) Analysis

The impregnated activated carbon is heated at 300˚ C for 2 h. The XRD spectrum for activated carbon rice husk biochar (AC-RHB) sample was shown in Figure 2. The XRD pattern for AC-RHB showed the amorphous nature although, it has some local crystalline structure with high conjugated aromatic elements. From the result, XRD patterns revealed the absence of any ordered crystalline structure and the broad band localized between (20° and 60°) 2θ corresponded to the contribution from both amorphous carbon and silica.

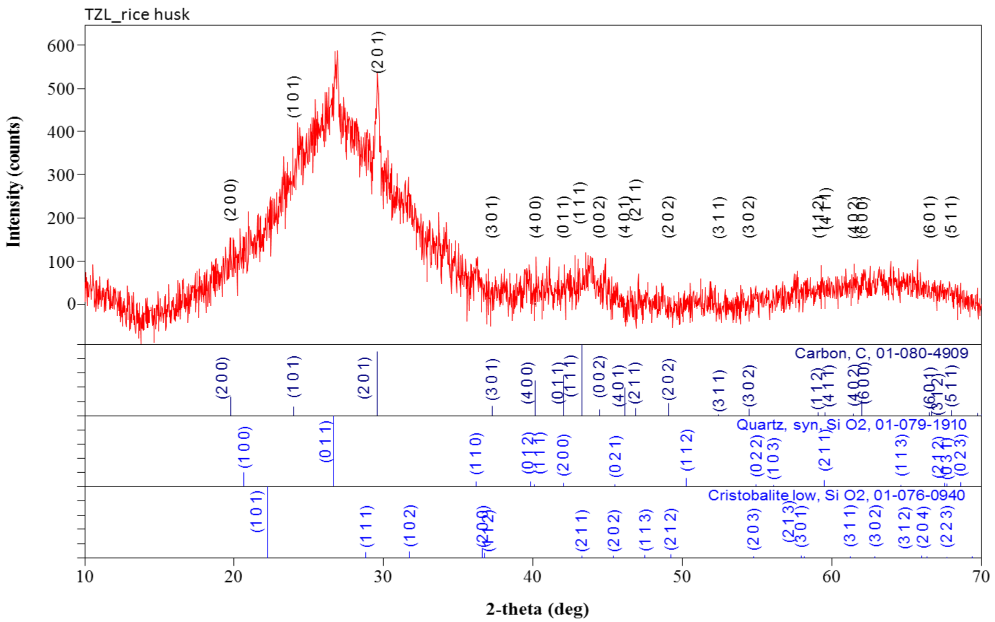


Figure 2: XRD spectrum of AC-RHB at 300˚ C

Energy Dispersive X-ray Fluorescence (EDXRF) Analysis

Table.2 indicates that elemental concentration in AC-RHB at 300˚ C for 1 h. Eleven elements namely C, K, Si, Ca, S, Fe, Cr, Cu, Mn, Ti and Ni were quantified in the rice husk biochar sample by EDXRF technique. Carbon (C) was found to be present as the major element, whereas K, Si, Ca, S, Fe, Cr, Cu, Mn, Ti and Ni were present as minor element. From the result, AC-

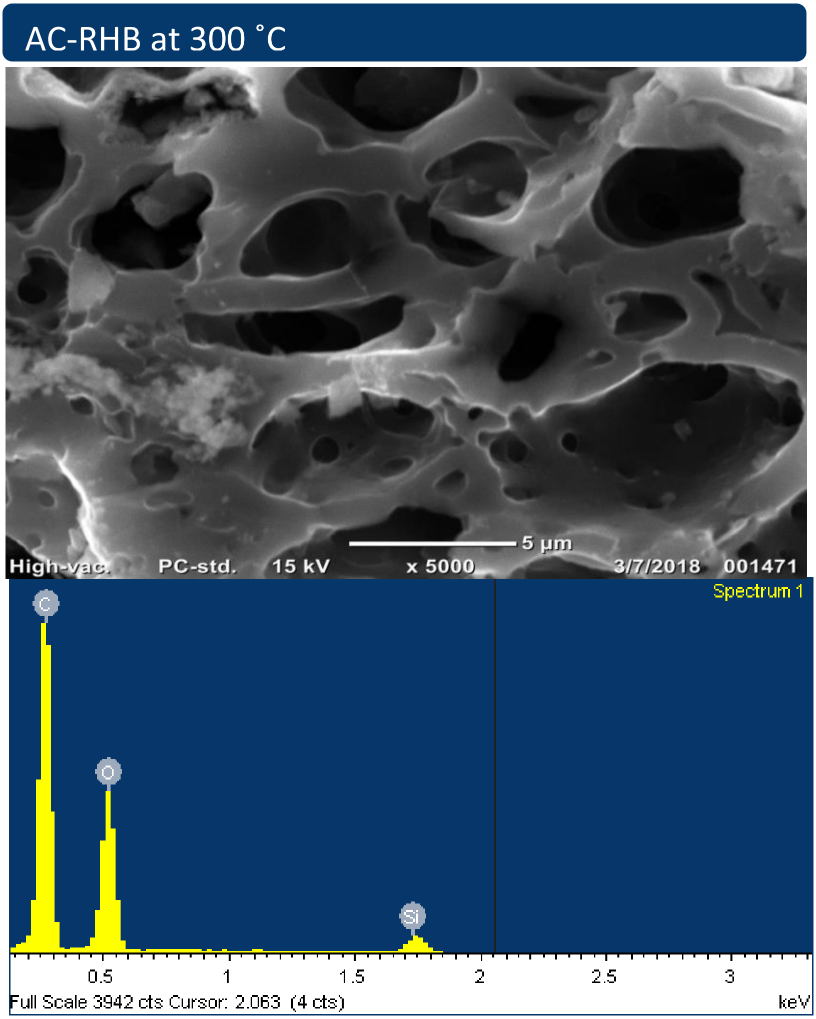
RHB contains 98.464 % of Carbon (C). Therefore, AC-RHB was quantified by EDXRF.

Table 2: Elemental Analysis of rice husk biochar

Elemental Analysis of AC-RHB												
Sr No.	**Element**	**C**	**K**	**Si**	**Ca**	**S**	**Fe**	**Cr**	**Cu**	**Mn**	**Ti**	**Ni**
1.	Content Weight %	98.464	1.226	0.224	0.016	0.016	0.08	0.02	0.01	0.01	0.01	0.01

Scanning Electron Microscopy (SEM-EDX) Analysis

The microstructural properties of AC-RHB were observed by SEM analysis. Scanning electron micrographs for external morphology (SEM-EDX) analysis of AC-RHB at 300˚ C for 1 h were shown in Figure 3. At 300˚ C, the pores of about 5μm diameter were found on some region. From SEM micrograph, it was found that AC-RHB had irregular, rough and highly porous. The energy dispersion X-ray spectroscopy (EDX) corresponding to AC-RHB at 300˚ C for 1 h indicated that the AC-RHB has more minals C, O and Si. The elemental constitution of AC-RHB at 300˚ C with two major peak and one minor peak were found to have weight percentage of 71.81 of Carbon, 25.68 of oxygen and 2.51 of Silica. The RHB have atomic percentage of 74.32 of Carbon, 24.33 of Oxygen and 1.35 of Silica. It contains low fractions and absent of various elements such as Cl, K, S, Zn and Mg. This is important to study the influence of these impurities on the DCFC performance because the accumulated ashes would affect the life time of DCFC fuel source, high Carbon (C) content and low Sulphur content were required.



Element	**Weight%**	**Atomic%**
C	71.81	74.32
O	25.68	24.33
Si	2.51	1.35
Totals	**100.00**	

Figure 3: SEM and EDX spectrom of AC-RHB at 300˚ C

Fourier Transform Infrared Spectroscopy (FTIR) Analysis

FTIR result for 300˚ C of AC-RHB was shown in Figure 4. The first peak at 3231.01cm^{-1} represents the compounds with alcohol and phenolic groups having O-H stretching. The second peak that was located at 1559.37cm^{-1} indicates C=C stretching vibration consists of aromatic groups in lignins. The next peak that was located at 1361.02 cm^{-1} indicates presence of alkanes group having C-H stretching. Additionally, the peak at 1006.89 cm^{-1} corresponding to C-O stretch of alcohols, carboxylic acids, ester and ethers groups. The presence of functional groups such as the alcohol and aromatics groups, carboxylic acids, ester and ethers groups suggest that this AC-RHB could be affected in DCFC.

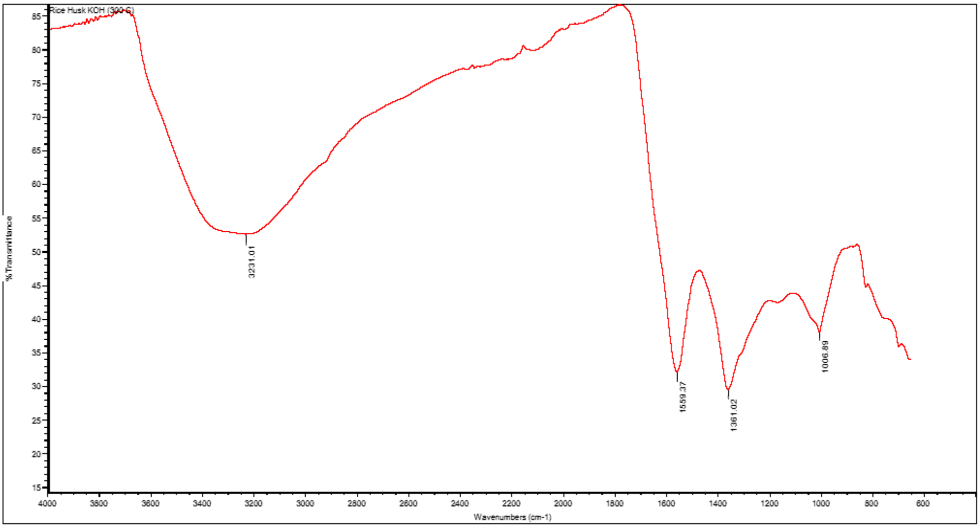


Figure 4: FTIR spectrum of AC-RHB at 300˚ C

Table 3: FTIR Analysis of AC-RHB

Sr No.	**Chimical bond**	**300°C Peak position (cm^{-1})**
1.	O-H Stretch	3231.01
2.	C=C Stretch	1559.37
3.	C-H Stretch	1361.02

4.	C-O Stretch	1006.89

Conclusion

In this work, biochar was successfully produced from rice husk using KOH treatment for activated carbon. The structural characterization of activated carbon rice husk biochar (AC-RHB) by X-ray Diffraction (XRD) technique revealed the presence of Carbon mainly and traces of SiO_2 were found. The treated RHB was quantified by EDXRF spectrometer. Carbon (C) was found that the highest concentration of about 98.464% in the AC-RHB. According to (SEM) analysis, AC-RHB which shows plane cleavage surface and irregular pores are observed. These results are good agreement with FTIR result. According to these results, AC- RHB (300^{o}C) is quite potential candidate for direct carbon fuel cell (DCFC) application.

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West Yangon University

Chemistry Department

EDX Report

Report No.

Sample Information

Sample Name	RH-KOH(300 C)
Meas. Date	2018/02/27 12:07:56
Comment	Quick&easy Air-Metal
Group	easy vacu
Operator	Dr Cherry Ohn

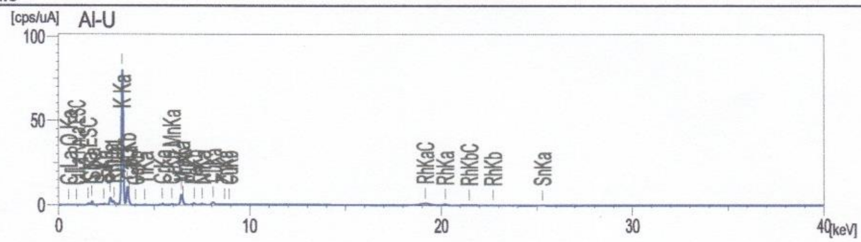
Measurement Condition

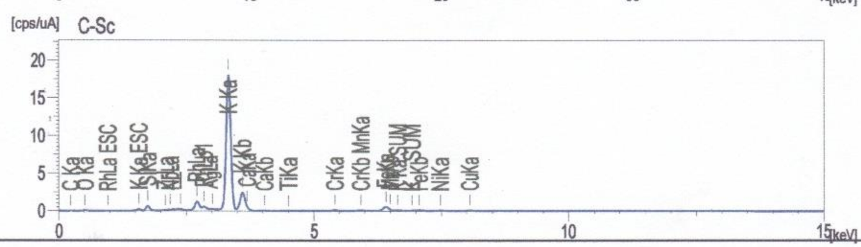
Channel	kV	uA	Filter	Acq.	Collimator 10mm Analysis	Time	Atmos. Vac. DT%
Al-U	50	61-Auto	----	0 - 40	0.00-40.00	Live- 30	30
C-Sc	15	193-Auto	----	0 - 20	0.00- 4.40	Live- 30	29

Quantitative Result

Analyte	Result		Std.Dev.	Calc.Proc	Line	Intensity
C	98.464	%	[93.135]	Quan-FP	C Ka	0.0524
K	1.266	%	[0.004]	Quan-FP	K Ka	199.1417
Si	0.224	%	[0.004]	Quan-FP	SiKa	5.5318
Ca	0.016	%	[0.000]	Quan-FP	CaKa	3.2490
S	0.016	%	[0.003]	Quan-FP	S Ka	3.9094
Fe	0.008	%	[0.000]	Quan-FP	FeKa	44.4989
Cr	0.002	%	[0.000]	Quan-FP	CrKa	5.8013
Cu	0.001	%	[0.000]	Quan-FP	CuKa	12.0924
Mn	0.001	%	[0.000]	Quan-FP	MnKa	3.8297
Ti	0.001	%	[0.001]	Quan-FP	TiKa	0.8234
Ni	0.001	%	[0.000]	Quan-FP	NiKa	4.9592
Sn	0.000	%	[0.000]	Quan-FP	SnKa	0.7118
Zn	0.000	%	[0.000]	Quan-FP	ZnKa	2.6710
O	0.000	%	[0.000]	Quan-FP	O Ka	0.5468

Profile





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