

PHYSICOCHEMICAL CHARACTERISTICS OF ACTIVATED CARBON FROM RICE HUSK BIOCHAR

Zin Min Tun¹, Zin Min Myat², Kyaw Zay Ya², Yin Maung Maung², Naw Htoo Lar Phaw¹

Abstract

Rice husk is an agricultural waste abundantly available in rice-producing countries. In this work, biochar is a solid material that is produced by heat decomposition of rice husk biomass by well-known pyrolysis method. Activated carbon was prepared by the conventional carbonization and KOH activation on a laboratory scale. The sample was calcined at 200 °C, 300 °C and 400 °C for 2 h to obtain biochar. The carbon structure of rice husk biochar activated carbon was examined by XRD. The chemical properties of the rice husk biochar activated carbon were confirmed by FTIR. The morphology of rice husk biochar activated carbon was determined by SEM. From FTIR analysis, three functional groups were found. According to SEM results, the average pore sizes of the samples were found to be about 6.15 µm at 200 °C, 5.80 µm at 300 °C and 4.13 µm at 400 °C respectively.

Keywords- Rice Husk biochar, Activated carbon, XRD, FTIR, SEM

Introduction

Biochar is charcoal used as a soil amendment for both carbon sequestration and soil health benefits. Biochar is a stable solid, rich in carbon, and can endure in soil for thousands of years [Lean, Geoffrey, 2011]. Biochar is produced by thermal decomposition of biomass under oxygen-limited condition (pyrolysis), and it has received attention in soil remediation and waste disposal in recent years. The characteristics of biochar are influenced mainly by the preparation temperature of biomass. Higher pyrolysis temperature often results in the increased surface area and carbonized fraction of biochar leading to high absorption capability for pollutants. Biochars derived from various source materials show different properties of surface area porosity and the amount of functional groups which are important concerning on the effect of biochar [Jingchun Tang, et al, 2013]. Biochar has been proved to be effective in improving soil properties and increasing crop biomass. It has also been suggested that it can even enhance crop resistance to disease. Biochar has recently been used to remediate soil with both heavy metal and organic pollutant [Jingchun Tang, et al, 2013, Luke Beesley, et al, 2011]. In addition to its potential for carbon sequestration and decrease greenhouse gas emission from agriculture, biochar is reported to have numerous benefits as a soil amendment, increased plant growth yield, improved water quality, reduced leaching of nutrients, reduced soil acidity, increased water retention and reduced irrigation and fertilizer requirements [Wen Wang, et al, 2013]. Bio-char is a stable solid, rich in carbon and can endure in soil for thousands of years. Biochar carbon species vary in complexity from graphite like carbon to high molecular weight aromatic rings, which are known to persist in soil for thousands to millions of years [Mohamad Azri Sukiran, et al, 2011].

Activated carbon, also called activated charcoal, is a form of carbon processed to have small, low-volume pores that increase the surface area [Bulletin of the American Physical Society, 2013] available for adsorption or chemical reactions [CPL Caron Link, 2008]. Activated is sometimes substituted with active. Due to its high degree of microporosity, one gram of activated carbon has a surface area in excess of 3,000 m² (32,000 sq ft) [Dillon, et al, 1989] as determined by gas adsorption [P. J. Paul.]. An activation level sufficient for useful application may be obtained

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solely from high surface area. Further chemical treatment often enhances adsorption properties. Activated carbon is usually derived from charcoal and is sometimes used as biochar.

Materials and Method

The rice husks (RH) were cleaned with deionized water to remove the dust and impurities. These RHs were dried in sun for one day and then hand crushed to smaller pieces. Then, the smaller pieces of these RHs were dried at room temperature about one week. The weight of rice husks were measured with digital balance. And then, these raw RHs were carbonized in the muffle furnace at three different temperatures, which were 200 °C, 300 °C and 400 °C for 2 h respectively. After carbonization, these rice husk biochars were mixed with deionized water and KOH in a stainless steel beaker with weight ratio of KOH/RH biochar equal to 1:2. Water was evaporated at 120 °C for 6 h, and these dried mixtures were heated in the muffle furnace at 800 °C for 1 h. The products were cooled to room temperature and washed with HCl and deionized water until the pH of the washing solution reached 6-7. Finally, the activate carbon obtained from rice husk biochars. The carbon structure confirmation of rice husk activated carbon (RHAC) was confirmed by X-ray Diffractometry (XRD). The morphology and microporous structures of RHAC were determined by Scanning Electron Microscopy (SEM, JEOL 6000) and the chemical properties of biochars were examined by Fourier Transform Infrared Spectroscopy (FTIR, Thermo Scientific). The block diagram for experimental procedure of rice husk biochars was shown in Figure 1. Table 1 showed the weight loss and weight loss % of rice husk biochars.

Table 1 Weight loss and weight loss % of rice husk biochar

Temperature	W ₁	W ₂	Weight loss, W = W ₁ – W ₂	Weight loss %
200 °C	5.86 g	5.02 g	0.84 g	14.33 %
300 °C	5.89 g	3.36 g	2.53 g	42.95 %
400 °C	6.00 g	3.14 g	2.86 g	47.67 %

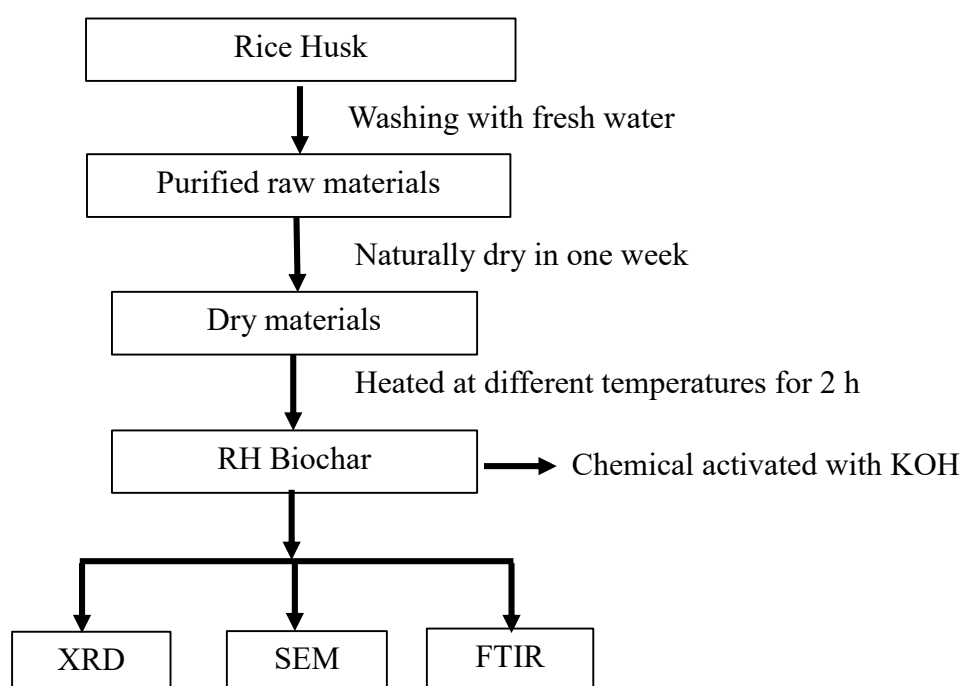


Figure 1 The block diagram for experimental procedure of coconut and peanut shell biochars

Results and Discussion

FTIR Analysis of Rice Husk Biochars

FTIR spectroscopy was applied to measure the chemical properties and absorption of energy from the range of 4000 cm^{-1} - 500 cm^{-1} by studied samples. Spectral registration was examined with use of solid-state samples which is made of a complex organic material. The FTIR analysis demonstrated the functional groups presented on rice husk biochar at different process temperatures. The functional groups of rice husk biochar have found to be O-H stretching vibration, C=O stretching vibration and C-O stretching vibration respectively. The spectrum of these samples showed some characteristic bands related to physical and chemical changes. As shown in Figure 2(a-c), the infrared spectra of these biochar types are comparable but there are some changes in the functional groups. The water O-H stretch can occur in the rice husk biochar for about 3324.85 cm^{-1} , 3240.00 cm^{-1} and 3236.43 cm^{-1} at three different temperatures. The absorption bands, between 3000 cm^{-1} - 3300 cm^{-1} indicated the presence of strong carboxylic acid O-H stretch. As observed peak of RH biochar, C=O stretching is associated with peak values 1599.15 cm^{-1} , 1601.15 cm^{-1} and 1637.85 cm^{-1} respectively. The presence of the band located at 1028.45 cm^{-1} - 1073.68 cm^{-1} showed the strong C-O stretching modes. According to FTIR analysis, all of the absorption bands are due to hydroxyl group in cellulose, carbonyl groups of acetyl ester in hemicellulose, and carbonyl aldehyde in lignin. Table 2 showed the FTIR analysis data of rice husk biochars at different temperatures.

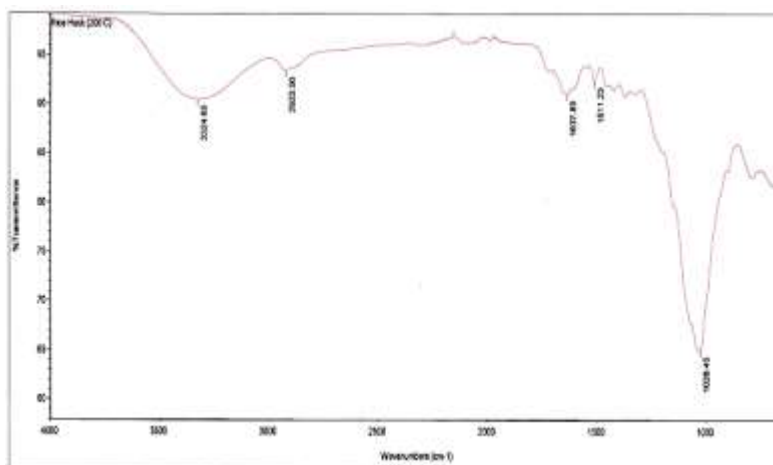


Figure 2(a) FTIR spectrum of rice husk biochar at $200\text{ }^{\circ}\text{C}$

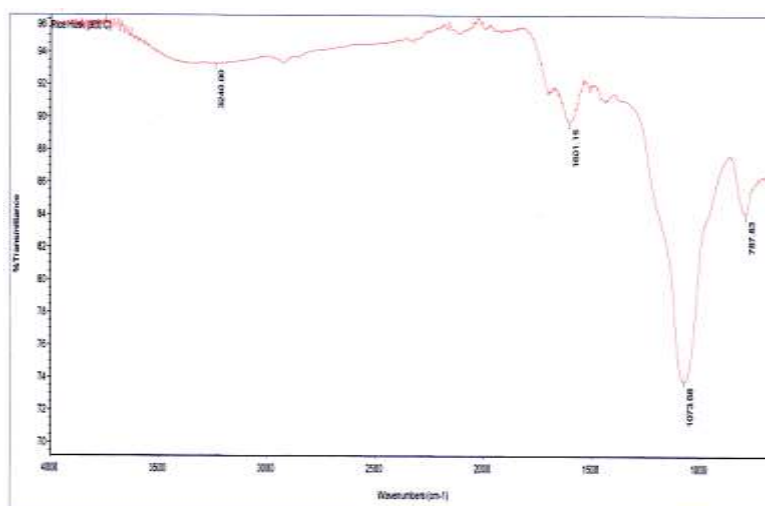


Figure 2(b) FTIR spectrum of rice husk biochar at $300\text{ }^{\circ}\text{C}$

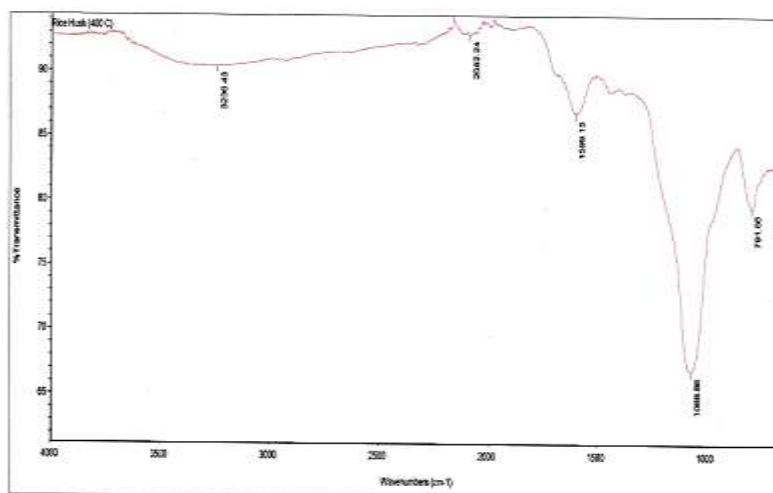


Figure 2 (c) FTIR spectrum of rice husk biochar at 400 °C

Table 2 FTIR analysis data of rice husk biochars at different temperature

Temperature (°C)	Wave numbers (cm ⁻¹)	Functional group
200	1637.85	C=O stretching
	2922.00	C-H stretching
	3324.85	O-H stretching
300	1073.66	C-O stretching
	1601.15	C-O stretching
	3240.00	O-H stretching
400	1599.15	C=O stretching
	2082.24	C-H stretching
	3236.43	O-H stretching

SEM Analysis of Rice Husk Biochars

SEM is one of the most versatile instruments available for the examination and analysis of the microstructure characteristics of a solid. The most important reason for using SEM is high resolution that can be obtained when bulk sample are examined. SEM micrographs for external morphology of rice husk biochar at temperatures 200 °C, 300 °C and 400 °C for 2 h were shown in Figure 3(a-c). According to Figure 3(a-c), the clear porous nature had observed the rice husk biochar at 200 °C, 300 °C and 400 °C. After increasing temperature, it was found that the rice husk biochar had more clearer porous nature and uniform with microporous structure. From SEM analysis as shown in Figure 3(a-c), it can be observed that the microstructure of rice husk biochar samples by varying the pore sizes with different temperatures. At 200 °C, the RH biochar had uniform porous nature and had large surface area. At 300 °C, the pores of biochar were non-uniform and not clear. At 400 °C, the pores were more uniformly microporous structure. For RH biochar, the average pore sizes of the samples were found to be about 6.15 μm at 200 °C, 5.80 μm at 300 °C and 4.13 μm at 400 °C respectively.

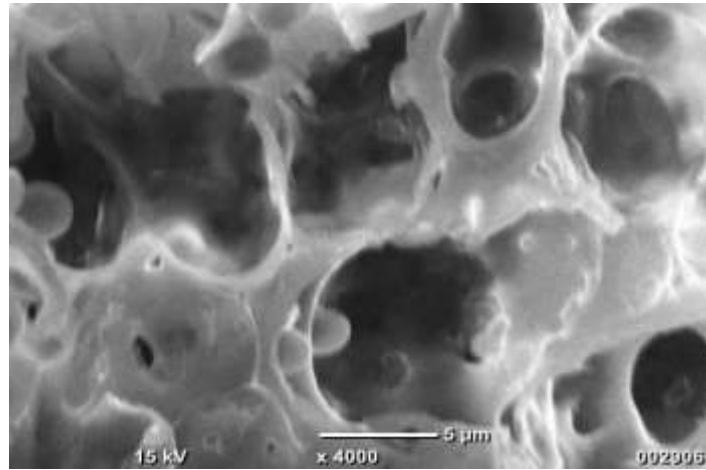


Figure 3(a): SEM micrograph of rice husk biochar at 200°C, (6.15 μm)

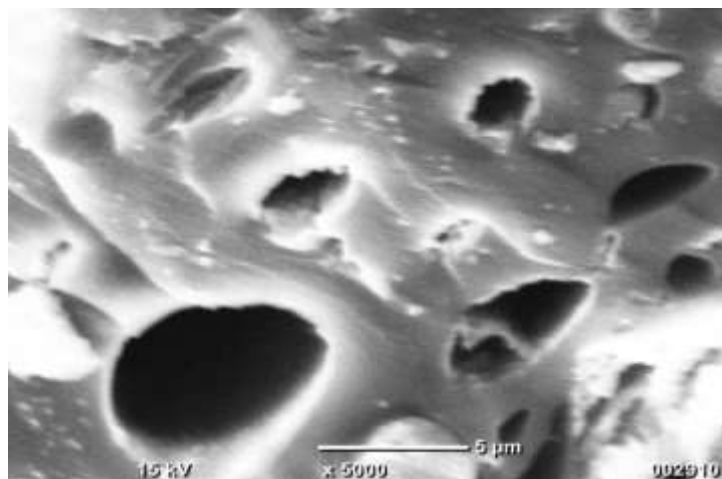


Figure 3(b): SEM micrograph of rice husk biochar at 300°C, (5.80 μm)

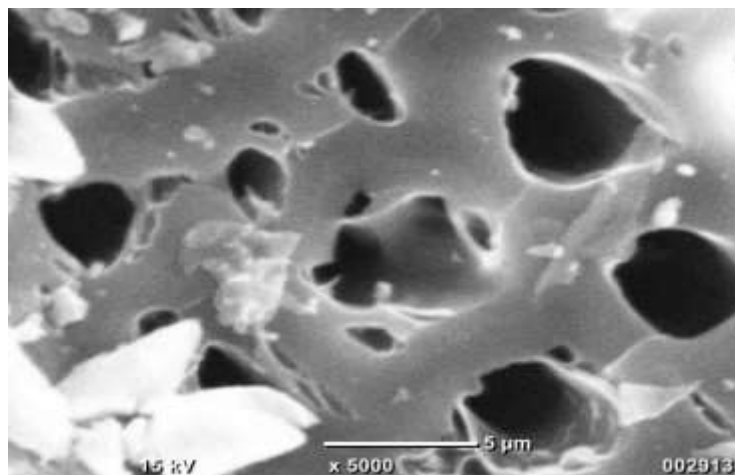


Figure 3 (c) SEM micrograph of rice husk biochar at 400°C, (4.13 μm)

X-ray diffraction analysis

X-ray diffraction is to determine the structure properties of rice husk biochar using monochromatic CuK α radiation ($\lambda = 1.54056 \text{ \AA}$) operated at 40 kV (tube voltage) and 40 mA (tube current). Analysis of rice husk biochar activated carbon at different temperature values (200 °C, 300 °C and 400 °C) for 2 h were shown in Figure 4(a-c).

From XRD results, these biochar exhibit the dominant diffraction peak located at around $2\theta = 20^\circ - 30^\circ$ that revealed the presence of amorphous structure which was disorderly stacked up by carbon rings. The XRD patterns of the rice husk biochar activated carbon showed the asymmetric (111) peak and (110) peaks maxima which were characteristic of graphite and carbon structures.

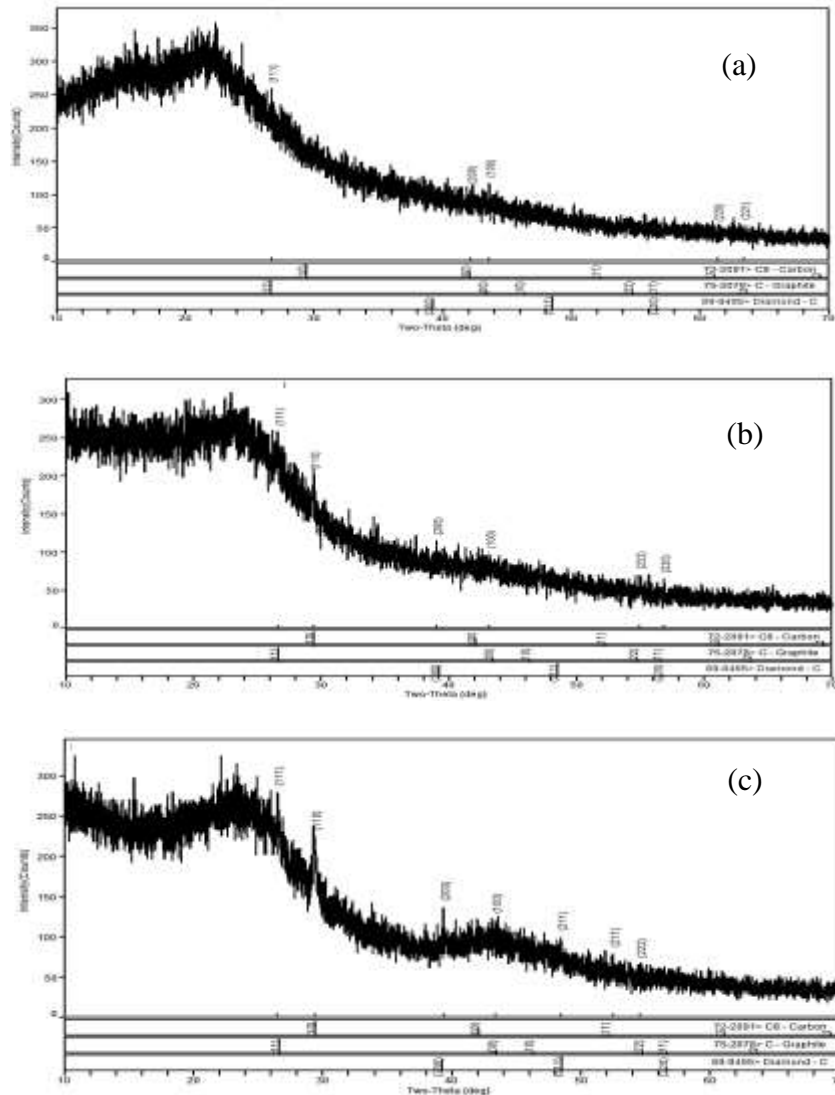


Figure 4(a-c) XRD patterns of Rice husk biochars activated carbon at different temperatures

Conclusions

In this research work, it was found that the weight loss % of RH biochar at different temperatures were 14.33%, 42.95% and 47.67% respectively. From the result, it can be observed that the weight and weight loss % of RH biochars were dependent on the calcinating temperature. The weight loss and weight loss % of RH biochars decreased with increasing calcinating temperatures.

For the results of FTIR analysis, all of the absorption bands are due to hydroxyl group in cellulose, carbonyl groups of acetyl ester in hemicellulose, and carbonyl aldehyde in lignin.

From SEM analysis, it can be observed that the porous structure of rice husk biochar samples by varying the pore sizes with different temperatures. For RH biochars, the average pore

sizes of the samples were found to be about 6.15 μm at 200 °C, 5.80 μm at 300 °C and 4.13 μm at 400 °C respectively.

As the results of XRD analysis, there are several diffracted peaks were observed. They were not perfectly identified. It could be say that the RHAC samples were found to be amorphous structure with little crystalline. XRD patterns of RHAC were quite acceptable. Almost all the reflections were found to be consistent with carbon.

According to these analyzed results, rice husk was calcinated at 200 °C, 300 °C and 400 °C to get the rice husk biochar, a pozzolanic material, which can be used in partial replacement of cement and soil amendment for agriculture. These rice husk activated carbon can also be used in removal of heavy metal from waste water solution for environment.

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References

- "Activated carbon monoliths for methane storage" (<http://meetings.aps.org/Meeting/MAR12/Session/W33.12>). Bulletin of the American Physical Society. 57 (1). 2012-03-01.
- "Adsorbed Methane Film Properties in Nanoporous Carbon Monoliths" (<http://meetings.aps.org/Meeting/MAR13/Event/186324>). Bulletin of the American Physical Society. 58 (1). 2013-03-20.
- Dillon, Edward C; Wilton, John H; Barlow, Jared C; Watson, William A (1989-05-01). "Large surface area activated charcoal and the inhibition of aspirin absorption". *Annals of Emergency Medicine*. 18 (5): 547–552.
- Jingchun Tang , Wenying Zhu, Rai Kookana, Arata Katayama, "Characteristics of Biochar and Its Application in Remediation of Contaminated Soil", *J Biosci Bioeng*, Dec. 2013,116(6),653-9.
- Lean, Geoffrey, "Ancient skills' could reverse global warming", *The Independent*. October 2011.
- Luke Beesley, Eduardo Moreno-Jiménez et al, "A review of biochars' potential role in the remediation", *j.envpol*, 2011 Dec, 159(12), 3269-3282.
- Mohamad Azri Sukiran, Loh Soh Kheang, Nasrin Abu Bakar and Choo Yuen May, "Production and Characterization of Bio-Char from the Pyrolysis of Empty Fruit Bunches", *American Journal of Applied Sciences*, 8 (10): 984-988, 2011.
- P. J. Paul. "Value Added Products from Gasification–Activated Carbon", Bangalore: The Combustion, Gasification and Propulsion Laboratory (CGPL) at the Indian Institute of Science (IISc).
- "Properties of Activated Carbon", CPL Caron Link, accessed 2008-05-02".
- Wen Wang, Dominic C Moran, et al,"Economic Potential of Greenhouse Gas Mitigation Measures in Chinese Agriculture", *Report,UK-China, Sustainable Agriculture Innovation Network-SAIN*, July 2013.