



Effect of Heat treatment on the Chemical and Microstructural Properties of Wheat Straw Ash (WSA)

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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ABSTRACT

This study examines the effects of different burning temperature on the chemical and microstructure properties of WSA. In many parts of the world due to non-availability of proper technology, the farmers generally burn wheat straw after acquiring grains, which causing environmental pollution with fire hazards at farm level and loss valuable commodity. In this research, the influence of different temperature on locally available Wheat Straw in Province KP, Pakistan was study. This research aim to find optimum temperature and burning duration of WSA by examine chemical and microstructural properties of WSA at 550°C for 4 hr, 550°C for 8 hr and 800°C for 30 min. The tests results were categorized through X-ray Diffraction (XRD), X-Ray fluorescence (XRF) and Fourier Transform Infrared Spectroscopy (FTIR). The result showing the 550°C for 4 hr contains high amount of (SiO₂+Al₂O₃+Fe₂O₃) and amorphous nature as compared to other samples.

Keywords: *Wheat Straw Ash (WSA); calcination; optimum temperature; micro-structure properties; amorphous nature.*

1. INTRODUCTION

A significant amount of waste material is produced worldwide as by-products from various sectors, for instance, waste from industrial, agricultural, rural and urban societies. This waste material can be harmful if not placed carefully. An increase in population leads to an increase in different types and amounts of waste. This residue remains in the environment longer because it is not used. Due to large amount of these wastes present in the environment, it has arisen the dumping crisis. The solution to this crisis is to reutilize the waste into valuable outcomes. Research on inventive consumptions of waste is still progressing. Waste like silica fume, fly ash and rice husk ash have been used in concrete effectively [1].

Global population growth has led to an increase in demand for agricultural products such as wheat, rice, sugarcane, etc. Simultaneously, the increase in agricultural waste contributes to pollution in rural areas. Resource limitations and environmental pollution have tested the sustainable development of agriculture [2,3]. Agricultural waste produced is directly disposed of without any technical processing. It is essential to use these waste, by reducing pollution and also increase economic assistances [4,5]. To ensure the sustainable development of agriculture farms, agricultural waste can be converted into valued assets through reutilizing [6].

Wheat straw ash is one of the interesting and attractive materials for researchers to be used as supplementary cementitious material (SCM). Wheat is a cereal crop, which produces wheat straw as an agriculture waste. This wheat straw after burning in the open field by farmer produces CO₂ and causes environmental pollution.

Wheat straw burned at a controlled temperature contains an enormous amount of silica.

The silica in WSA gives it a pozzolanic property, which can be utilized in concrete as SCMs.

There are several benefits of using WSA as SCMs, like reduction in CO₂ emission and also a solution for agricultural waste product that is obtained from the production of cereal. Farmers burnt WSA in open fields which contributes to

environmental pollution.. Using WSA in concrete will have an economical solution for the construction industry that will have a optimistic effect on the properties of concrete [7].

In Pakistan wheat is one of the main agriculture crops. The global annual wheat production is around 880 million tons, while in Pakistan the annual production is approximately 25 to 28 million tons [8]. Most of the wheat straw produce worldwide were used for cattle food. Around 1.3 kg to 1.4 kg wheat straw is produced per 1 kg of wheat grains [9]. In most of the cases wheat straw is burnt in the open field in most part of the world as well as in Pakistan. Which causing environmental pollution by producing CO₂ having negative impacts on environment and as a result greenhouse effect. On the other side after burning wheat straw in the open field the ash remaining, causes damping problem, creating health issues for the residents living near to that area. In recent times research show that agriculture by-products such as Wheat Straw Ash (WSA) are pozzolanic in nature and use as supplementary cementitious material as a cement replacement in concrete after burning and grinding at optimum temperature [10].

Several researchers have conducted research studies to utilize Wheat Straw Ash (WSA) as a Supplementary Cementitious Materials (SCMs) as a replacement of cement in concrete and mortar [11]. Investigations were carried out on burning wheat straw between 570°C and 670°C for duration of 5 hr, the WSA acquired by burning at 670°C showing more pozzolanic performance as related to 570°C burning [12]. Another research study was conducting by Wheat Straw Ash (WSA) at four distinct temperatures (500°C, 600°C, 700°C and 800°C) to check the pozzolanic behavior of WSA. The enhanced pozzolanic behavior was observed at 600°C, which is the optimum temperature. The results also showed that by increasing the burning temperature from 600°C to 800°C, the silica converts from amorphous to crystalline form [13].

Ataie et al. [14] WSA burnt at a control temperature produces more amorphous silica and reduces processing costs. They also investigated that when WSA burnt for duration of 12 hr and 1 hr at 500°C and 600°C respectively showed enriched amorphous silica content as related to burning at the same temperatures for

extended period. In fact, source is the key factor for the pozzolanic proficiency of WSA and also the type of soil and weather conditions of the area from which WSA obtained has impacts on the composition of WSA [15].

In this research work, the main objective is to study the influence of different combustion temperatures and duration on chemical and microstructure of WSA and also to check the amount of Silica, alumina and iron oxide ($\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$) in amorphous form. Wheat Straw were collect from Khyber Pakhtunkhwa, Pakistan. Burnt the dry wheat straw at three different temperatures and duration (550°C for 4 hr, 550°C for 8 hr and 800°C for 30 min) in rotary kiln, Pakistan Council of Scientific and Industrial Research (PCSIR) laboratory Peshawar, Pakistan. To analyze the chemical composition and microstructure properties, X-Ray Diffraction (XRD), X-Ray Fluorescence (XRF) and Fourier Transform Infrared Spectroscopy (FTIR) tests were conducted for each samples to investigate silica content and amorphous nature of WSA.

Successful use of WSA in today's environment offers a number of benefits, including a low-cost solution for the construction industry and a reduction in health and environmental problems.

2. MATERIALS AND METHODS

2.1 Materials

2.1.1 Collection, burning and grinding of wsa

Wheat Straw was collected from local field of Khyber Pakhtunkhwa, Pakistan. To remove any dust wheat straw was carefully washed before burning and then drying in the open air. Wheat Straw was burnt in Kiln at Pakistan Council of Scientific and Industrial Research (PCSIR) laboratory Peshawar at three different temperatures and duration of 550°C for 4 hr, 550°C for 8 hr and 800°C for 30 min in a rotary kiln and then grind in a ball mill for 16 hour to obtain finer WSA that passing through sieve #200 as shown in Fig. 1 and Fig. 2.

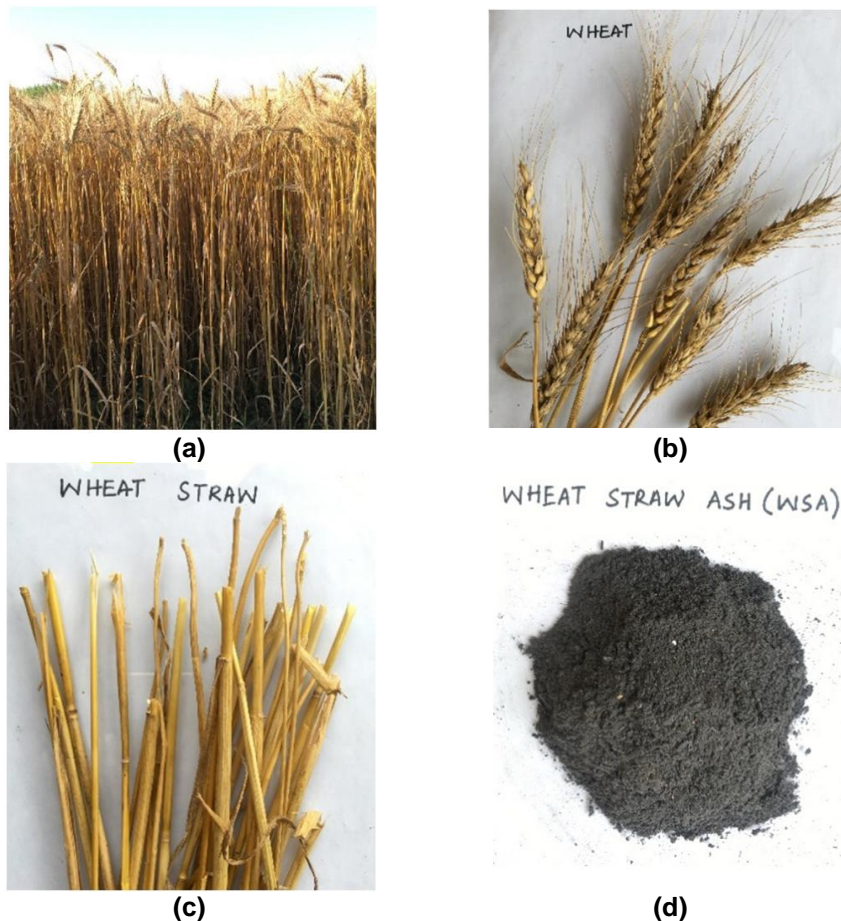


Fig. 1. (a) Wheat crop (b) Wheat Plant (c) Wheat straw and (d) Wheat straw ash

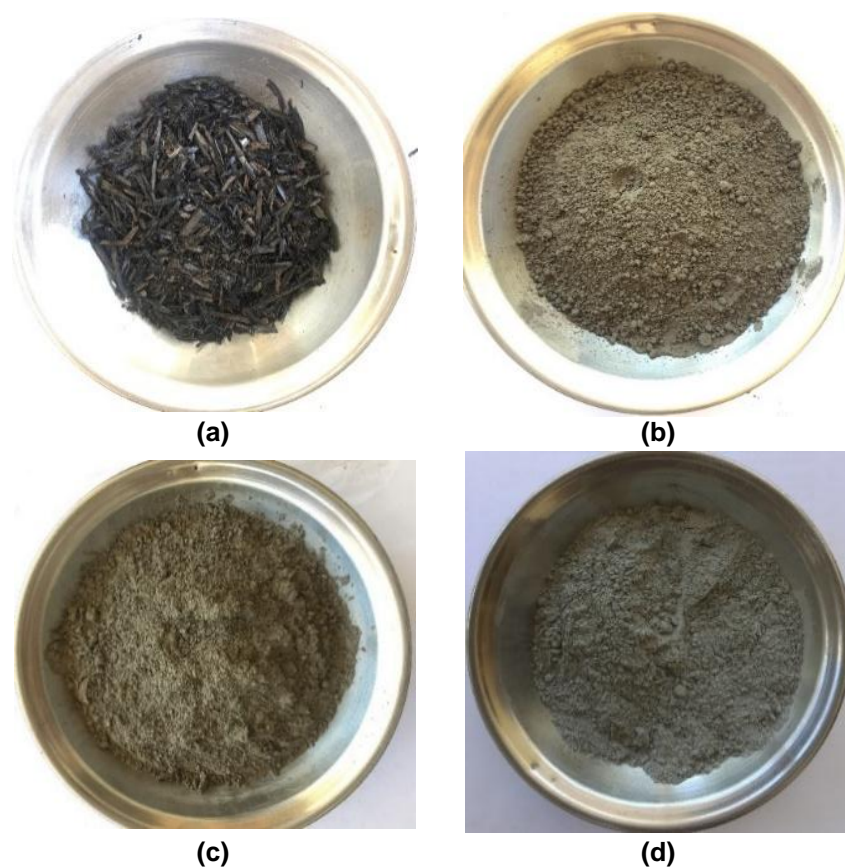


Fig. 2. (a) WSA before grinding (b) WSA burning at 550°C for 4 hr (c) WSA burning at 550°C for 8 hr (d) WSA burning at 800°C for 30 min

WSA was acquired by burning wheat straw at three changed temperatures i-e 550°C for 4 hr and 8 hr and 800°C for 30 min in a rotary kiln as shown in Fig. 3 and then grind in a ball mill for 16 hour as shown in Fig. 4. The byproduct of wheat crop is wheat straw and after burning this wheat

straw at optimum temperature, it contains enormous amount of SiO_2 . To determine the optimal temperature and period wheat straw was burnt at different temperatures at which WSA contains high amount of silica and also amorphous nature.



Fig. 3. Rotary Kiln used for burning WSA



Fig. 4. Ball Mill in which WSA grind

2.2 Methods

2.2.1 X-ray diffraction analysis & X-Ray fluorescence:

XRF and XRD analysis of WSA samples is very important for the reason that it gives information about the composition of elements and oxides, as well as the properties of these compounds, as amorphous /crystalline structures found in WSA. The XRF analysis give the elemental and oxides composition of WSA while the XRD define the crystalline nature of the WSA.

To find chemical composition about elements, oxide and their properties, XRF and XRD tests were performed on WSA samples at the Materials Research Laboratory, Physics Department University of Peshawar. Equipment details are given in Table 1.

Table 1. Equipment details

| | |
|----------------------|--------------------------------|
| Model No. | JDX – 3532 |
| Company | JEOL (Japan) |
| Potential Difference | 20-40 kV |
| Current | 2.2-30 mA |
| X-Ray | CuKa, Wavelength = 1.5418 Å |
| 2θ-Range | 0° to 160° |

This device works on the principle that each time, when a substance passes in an x-ray, it propagates it from changed angles dependent on its chemical configuration. When the material consists of a crystalline phase, a high intensity peak will be observed during XRD analysis. The

crystalline phase is categorized through narrow peaks, while the amorphous phase has a wider peak, but is less intense than the crystalline phase. Equipment is shown in Fig. 5.

2.2.2 Fourier transform infrared analysis (FTIR)

The qualitative analysis of WSA categorized using Fourier Transform Infrared Spectroscopy (FTIR) in order to define the variations in specific functional group of the different burn WSA. The equipment were used to study FTIR test is IRTracer-100 (Fourier transform Infrared Spectrometer, SHIMADZU) as shown in Fig. 6.

3. RESULTS AND DISCUSSION

3.1 X-Ray Diffraction Analysis

XRD analysis showing the qualitative analysis of silica, also shows the amorphous and crystalline phase in WSA. Amorphous silica is reported to be more reactive through pozzolanic reaction related to crystalline silica [16]. WSA chemical configuration indicates that major ingredients are SiO₂ and K₂O. There are many factors on which WSA chemical configuration depends such as the soil in which wheat plant is growing, different types of fertilizers used, environment of the cultivated area, burning temperature, volume of burning wheat straw and burning duration [17,18]. During wheat crop development the soil and environmental conditions affect the SiO₂ content in WSA [19]. Fig. 7 (a,b and c), showing XRD patterns of vary WSA. The XRD graph shows that the WSA is mostly amorphous

contains two phases which are cristobalite and quartz. For WSA 550°C at 4 hr a sharp peak which is wide at 2 theta, which recognized as quartz (SiO₂). However, the presence of silica is commonly in amorphous stage as related to 550°C 8 hr and 800°C for 30 min.

The quantity of amorphous stage was calculated using Rietveld refinement. The intended value ~86.9 mass % for WSA 550°C for 4 hr sample has shown the high amorphous nature of the investigated WSA. Given the high amorphous nature of WSA burned at 550°C for 4 hr, it is ground in a ball mill for 16 hours to attain the anticipated fineness essential for the pozzolanic material.

3.2 X-Ray Fluorescence (XRF)

To evaluate the effect of different combustion temperatures (550°C for 4 hr and 8 hr and 800°C for 30 min duration) on WSA, XRF analysis were performed to find its chemical composition which are specified in Table 2: From the table the amount of main oxides (SiO₂+Al₂O₃+Fe₂O₃ = 72.8%) optimum content were obtained for 550°C at 4 hr as compared to other two burning temperatures i-e 550°C 8 hr and 800°C for 30 min duration were 70.5% and 63.53% correspondingly. The silica content in sample (550°C 8 hr) was in enormous amount, but from XRD test most of the silica has been converted from amorphous to crystalline form.



Fig. 5. XRD and XRF equipment



Fig. 6. IRTracer equipment for finding FTIR

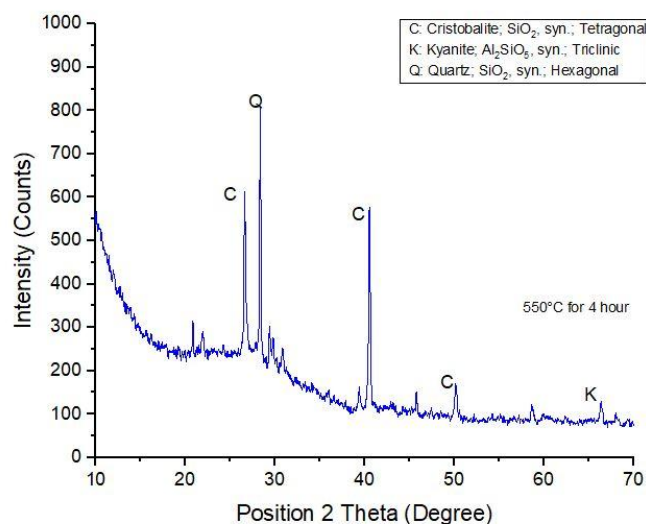


Fig. 7 (a). XRD pattern of WSA at 550°C for 4 hour

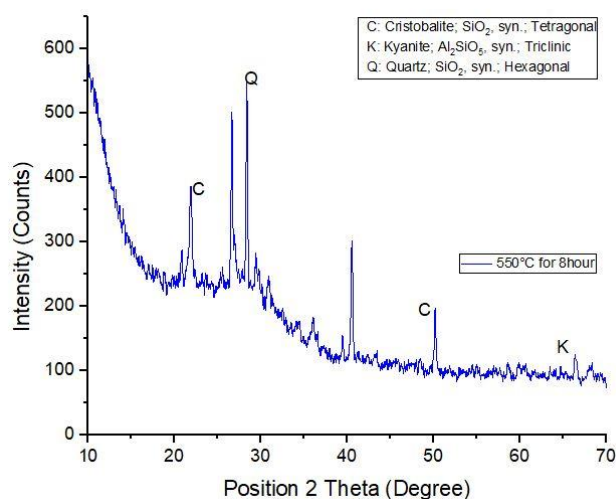


Fig. 7 (b). XRD pattern of WSA at 550°C for 8 hour

3.3 Fourier Transform Infrared Analysis (FTIR)

Fourier Transform Infrared Spectroscopy of WSA was performed at different burning temperatures in powder form in Laboratory of US-Pakistan Center for Advanced Studies in Energy (USPCAS-E), UET Peshawar. The results are shown in Fig. 8. The FTIR spectra of WSA (550°C for 4 hr and 8 hr and 800°C for 30 min duration). The infrared (IR) sample band is important with the possibility of its transfer are given in Table 2. By equating the detected wavelengths through the existing literature, minerals for example quartz, orthoclase, kaolinite and montmorillonite have been recognized.

Peaks looking in the range 2850-2853 cm^{-1} and 2923-2926 cm^{-1} indicate the presence of organic carbon in entirely samples [20]. Nitrate elements content showed that N-O is present in these samples. FTIR observations showed the quartz, feldspar (amorphous silica), montmorillonite and organics matter in WSA samples. The crystal structure showed in between the region 3800 cm^{-1} and 3000 cm^{-1} . This range includes the total vibrations of the valence band of hydrogen bonds of the O-H group and of the intramolecular band and intermolecular hydrogen bond [21].

The mineral groups, wavelengths and tentative assignment of the WSA are given the Table 3.

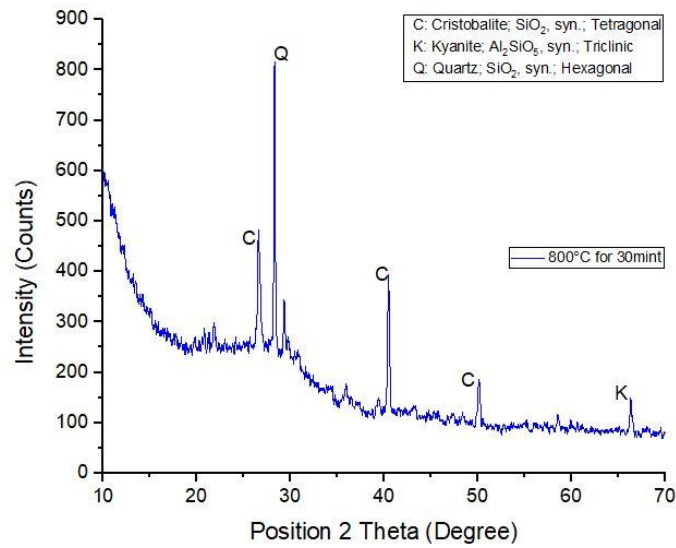


Fig. 7 (c). XRD pattern of WSA at 800°C for 30 min

Table 2. WSA oxide composition w.r.t. combustion temperatures

| Item | Oxides content (% by Weight) | | |
|--|-------------------------------------|-----------------------------|-------------------------------|
| | Combustion temperature and duration | | |
| | 550 ^o C for 4 hr | 550 ^o C for 8 hr | 800 ^o C for 30 min |
| SiO ₂ | 62.05 | 60.07 | 55.5 |
| Al ₂ O ₃ | 9.08 | 6.98 | 11.24 |
| Fe ₂ O ₃ | 1.67 | 1.45 | 2.79 |
| SiO ₂ +Al ₂ O ₃ +Fe ₂ O ₃ | 72.8 | 68.5 | 69.53 |
| K ₂ O | 17.05 | 15.87 | 22.43 |
| CaO | 5.90 | 8.47 | 7.40 |
| SO ₃ | 1.66 | 1.67 | 1.84 |

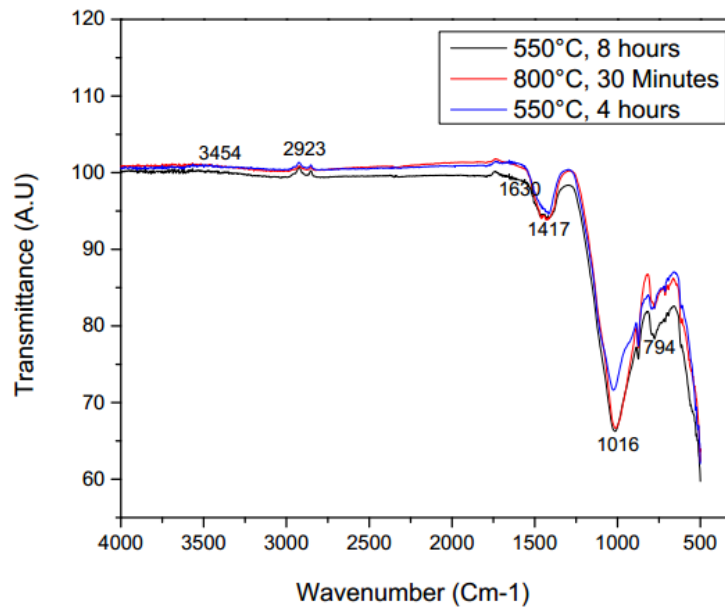


Fig. 8. FTIR Spectrum of WSA (550°C for 4 hr and 8 hr and 800°C for 30 min duration)

Table 3. Band assignment of WSA (550°C for 4 hr and 8 hr and 800°C for 30 min duration)

| Mineral component | Tentative assignment | Wavelengths (cm ⁻¹) | Reference |
|-------------------|---|---------------------------------|-----------|
| Montmorillonite | (OH) group adsorbed water dust-stretching | 3454 | [22] |
| Organic Carbon | (C-H) Stretching vibration | 2923 | [23] |
| Organic matter | C=O carboxylate stretching vibrations group of organic matter | 1630 | [24,25] |
| Nitrate Species | (N-O) Stretching | 1417 | [26] |
| Quartz | (Si-O-Si) asymmetric stretching vibration | 1016 | [27] |
| Quartz | (Si-O) symmetric | 794 | [28,29] |
| Felspar | Si-O-Si band (Amorphous silica) | 563 | [30] |

4. CONCLUSION

A local WSA in Khyber Pakhtunkhwa, Pakistan were examined to study its capability as sustainable building raw material through assessing its pozzolanic action. Following are the conclusions that are obtained from the investigation.

- XRD results concluded that the properties of WSA change from amorphous to crystalline as the temperature rises from 5500C to 8000C.
- WSA contains high mount of amorphous silica at 5500C for 4 hour as compared to temperature of 5500C for 8 hr and 8000C for 30 min.
- From FTIR test the wavelengths (cm-1) were almost same for all temperatures but for 5500C for 4 hr shown high amount of amorphous silica.

Based on the tests performed and summarizing the data accessed experimentally, the analyzed WSA shows a capable material for the cement production, where ordinary portland cement can partially replace the clinker. From an environmental and economic point of view, the reuse of burning waste products analyzed in the production of building materials related to sustainability problems is a very useful solution.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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