Pergamon

Cement and Concrete Research 29 (1999) 637–643

CEMENT AND CONCRETE RESEARCH

Study of pozzolanic properties of wheat straw ash

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Manuscript received 15 January 1998; accepted manuscript 15 December 1998

Abstract

As an agricultural product, wheat straw contains considerable amounts of SiO_2: When burned it leaves an ash very rich in SiO_2 that has a pozzolanic character. Wheat is an important agricultural product in Turkey. In this study, wheat straws are ground to 1–5-mm size and subjected to preburning treatment. The preburned material is later burned in controlled conditions for 5 hours at 570 and 670°C. The ash is cooled suddenly and ground to 90–200 μ size. The standard test specimens are produced from ash and mechanically, chemically, and physically tested for determination of its pozzolanic properties. It is obtained that the ash has pozzolanic activity.

Keywords: Pozzolan; Thermal treatment; Mechanical properties; Physical properties

The increase in the number of concrete types requires the use of new materials and technologies. Because of this fact, the types and quantities of cement production have been increased all over the world. To improve the properties and durability of concrete economically, the minerals having pozzolanic properties are also mixed with cement in concrete production. In addition to the natural pozzolanic materials, the industrial wastes (for example fly ash, slag blast furnace, and silica-fume) are also used as pozzolanic material. Some experimental studies are done to produce pozzolanic material from agricultural products. Pozzolanic materials are added to clinker during the production stage of cement or to cement for production of concrete. Pozzolanic materials are added to cement to fix the free lime released by clinker silicates during their hydration. This causes free lime to become insoluble in water, making the cement highly resistant to environmental effects. When a part of cement is replaced with pozzolanic material, the plasticity of concrete increases and the hydration heat of cement is reduced [1].

The amount of free lime combined by pozzolanic material is an indication of its pozzolanic property. This property depends greatly on the specific surface area of pozzolana. Other factors affecting the pozzolanic properties are amorphous SiO_2 or Al_2O_3 and SiO_2 content in the glassy or zeolitic phase. Pozzolanic materials are acidic type, therefore they are not soluble in water and oxides, except HF. Pozzolanic materials are of two types: natural and artificial [2,3].

Natural pozzolana consists of clays and sedimentary schists, opals and volcanic tuffs, and pumicite stones. They are found in certain places worldwide. Chemical composition and activity of pozzolana differ according to their locations. Specific gravity changes between 2000 and 2200 kg/m^3. Natural pozzolana are calcined in order to decompose carbonates to oxides [4].

Artificial pozzolana consists of calcined clay and some industrial wastes such as fly ash, slag, and silica fume. It contains SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, and other oxides. The amount of SiO_2 determines the activity of pozzolana. Industrial wastes are used to produce industrial pozzolana. Slag obtained from iron and steel industry, fly ash that is a by-product from coal-fired power stations, silica fume obtained from Si metal alloys, and ash obtained from other sources are such products. In addition to these, although not common, rice hull, wheat straw, and hazel nut shell are used as pozzolanic materials [3,5–7]. The following information concerns wheat straw as a pozzolanic material.

1. Production of pozzolana from agricultural wastes

Plants obtain various minerals and silicates from earth in their bodies during growth process. Inorganic materials, especially silicates, are found in higher proportions in annually grown plants than in the long-lived trees. Rice, wheat, sunflower, and tobacco plants therefore contain higher

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0008-8846/99/$–see front matter
PII: S0008-8846(98)00249-X

Table 1
Physical and chemical properties of cement

Physical properites	Mechanical properites	Days		
		2	7	28
Specific gravity (g/cm^3), 3.05	Flexural strength (N/mm^2)	4.8	6.1	7.60
Setting time, Initial (h), 1.50	Compressive strength (N/mm^2)	19	30.4	42.50
Setting time, Final (h), 3.35				
Le Chatelier soundness (mm), 3.0				
Specific surface area (Blaine)(cm^2/g), 3204				
Fineness-passing 200 μm (%), 0.20				
Fineness-passing 90 μm (%), 5.20				

amounts of silica in their cuticle parts. Inorganic materials are found in the forms of free salts and particles of cationic groups combined with the anionic groups of the fibers into such plants [8].

The burning of organic materials, production of new crystalline phases, or crystallization of amorphous material are exothermic processes that lead to ash production and loss in the total weight. The result of burning organic materials is called thermal decomposition [9]. The ash produced in this way is ground to a fine size and mixed with lime in order to obtain a material with a binding characteristic. The quality of this material depends on burning time, temperature, cooling time, and grinding conditions [10–12].

2. Pozzolanic activity experiments

In natural and industrial pozzolana, the pozzolanic activity is studied by using mechanical, chemical, and physical experiments. In the physical experiments, thermogravimetric analysis (TGA), differential-thermogravimeter (DTG), and diffractometric thermal analyses (DTA) are used [13]. Amorphous silica can be identified qualitatively by X-ray diffraction and quantitatively by chemical gravimetric methods [14]. In addition, reactivity of pozzolana can be determined rapidly by spectrophotometric and calorimetric methods. Thermodynamic instability, specific surface, and chemical composition are the parameters defining pozzolanic activity [3].

Table 2
Chemical analysis, modulus, and mineralogical compounds of cement

Chemical analysis (%)	Modulus	Mineralogical compounds (%)
SiO_2 (soluble) 20.60	Hydraulic mod., 2.09	C_3S* 40.18
Insoluble residue 0.38	Silicate mod., 2.09	C_2S 28.83
Al_2O_3 6.14	Aluminum mod., 1.65	C_3A 9.98
Fe_2O_3 3.72	Lime standard, 91.87	C_4AF 11.32
CaO 63.65		
MgO 1.29		
SO_3^- 2.55		
Loss on ignition 1.42		
Undetermined 0.25		
Free lime content (CaO) 2.08		

* S, SiO_2; C, CaO; A, Al_2O_3; F, Fe_2O_3; M, MgO; S^-, SO_4; H, H_2O.

2.1. Wheat and wheat straw

Wheat is the main agricultural product grown in Turkey and worldwide. It is estimated that world cereal production is about 880 million tons, of which 550 million tons is wheat straw. The amount of wheat straw production is 2.0–2.8 tons/ha [15]. The approximate amount of wheat straw is 19–27 million tons in Turkey, according to the State Institute of Statistics.

Wheat is a yearly plant that belongs to monovalve class in Gramineae family. Body (straw) is either simple or a group that is 35–100 cm in length, straight and completely bare or covered with bristles on the noodles. It grows on volcanic areas, hill slopes, steppes, and bare lands at various climates. The wheat plant has five main morphological parts: nods, internods, leaves, grains, and grain axis. The straws consist of C, H, O, N, Si, Fe, Al, Ca, Mg, Na, K, P, Cu, Mn, and Zn in various proportions. Straw has varying amounts of water, protein, oil, extractive material fiber, pentosan, cellulose, lignin, and ash. Leaves, guides, and nods contain high amount of silica. Proportions of the morphological parts vary depending on the wheat type, ecological conditions, and harvest height from ground [15–18].

2.2. Experimental work

Experimental work consisted of determining wheat straw properties, such as preburning stage, ash formation grinding, and pozzolanic properties. After the physical, morphological, and chemical properties were defined, optimum burning temperature and time were determined in order to obtain the ash with pozzolanic character. Physical and chemical properties of the ashes were studied. Ashes were mixed with cement and lime according to the standards specified.

Table 3
Physical and chemical properties of wheat straw

Physical properties	Chemical properties
Moisture (%), 9.54	Extractive substance (%), 4.23
Ash (%), 8.6	Ash (%), 8.60
Specific gravity (g/cm^3), 2.31	Lignin in carbohydrate (%), 15.03
Density (g/cm^3), 0.07	Holocellulose in carbohydrate (%), 45.13

Table 4
Physical properties of wheat straw ashes

Pozzolana	K5	K6
Specific gravity (kg/m^3)	2.31	2.41
Fineness (residue %)		
90 μm	5.4	2.6
200 μm	3.0	1.6
Specific surface Blaine (cm^2/g)	4850	5520

K5, ash production at 570°C; K6, ash production at 670°C.

2.3. Materials

Properties of cement, wheat straw, ash and sand are given as follows.

2.3.1. Cement

The cement was produced by Akçimento A.Ş (Istanbul, Turkey). The physical and mechanical properties of the cement are listed in Table 1 and the chemical properties of it are shown in Table 2.

2.3.2. Wheat straw

Wheat straw obtained from Tekirda-Kılavuzlu region has the physical and chemical properties shown in Table 3. X-ray diffraction pattern and scanning electron microscopy (SEM) photographs of it are given in Figs. 1 and 2, respectively.

2.3.3. Sand

Silica sand was used to produce mortar to determine the pozzolanic activities of the ash mechanically.

2.4. Ash production from wheat straw

Burning temperature and time were determined in the preliminary tests to produce ash from agricultural wastes having pozzolanic properties. To determine the optimum burning temperature, experiments were carried out at 300, 400, 500, 600, 700, 800, 900, and 1000°C. To determine the optimum burning times, experiments were also carried out for 1, 2, 3, 5, 8, 24, and 30 hours [10,12]. Variation of burning time depends on the thickness of specimen layer. Therefore burning time was determined experimentally [10].

To determine the ash content and SiO_2 content of straw, it was burned at 575 ± 25°C in electric furnaces for 5 hours and then cooled [19]. Before the burning the straws were collected and reduced to sizes of 1–5 mm and packed. It is shown that the ash content is 8.6% and SiO_2 content is 73–74%. According to Visvesvaraya, the ash content is 8–11% and SiO_2 content is 88–91% [14]. The difference in SiO_2 content depends on the soil content and environmental conditions.

The ash content also depended on the burning temperature and burning time. For example, although there was a complete burning at 800°C, some part of the amorphous structure was converted to a crystal structure [12,20]. The preliminary tests were carried out at 300, 350, 400, 450, 500, 570, 600, 670, and 700°C for various times longer than 3 hours. The suitable burning temperature was determined as 570 and 670°C for 5 hours. In this condition the gray and white color of the ash indicated a complete burning. At a burning temperature of 700°C, the ash showed a sintered structure.

The ash production was carried out in two stages as preburning and controlled burning. The preburning stage shown schematically in Figure 3 prevented smoke formation and reduced the total volume of wheat straw. It was burned in controlled conditions at 570 and 670°C for 5 hours in cages, using steel trays. At the end of each process, the furnace was cooled down to 300°C and the ash was taken out of the furnace and cooled to 20°C. The ashes produced at 570 and 670°C were observed having amorphous structure, as shown in Figs. 4 and 5 as X-ray diffraction patterns and in Figs. 6 and 7 as SEM examinations, respectively [7].

Table 5
Chemical properties of wheat straw ashes

Compound	K5 (%)	K6 (%)
SiO_2 (soluble)	50.78	54.24
SiO_2 (insoluble)	22.28	29.56
Al_2O_3	3.90	4.55
Fe_2O_3	1.75	1.05
CaO	8.12	12.54
MgO	2.80	2.39
SO_3	1.91	1.49
K	5.85	—
Na	1.83	—
Ca	3.05	—
Loss on ignition	8.79	7.22
Undetermined	—	—
Silica module	9.69	9.69
Aluminum module	4.33	4.33

Table 6
Material quantities of the mortars for pozzolanic activity

Material	Standard values	K5 (g)	K6 (g)
Standard sand	1350g	1350	1350
Hydrated lime	150 g	150	150
Pozzolan	$2 \times 150\ \delta p/\delta k$	315	329
Water	$0.5 \times (150+\text{pozzolan})$	395	325

Table 7
Mechanical test results and standard values

Pozzolan	Flexural strength (N/mm^2)	Compressive Strength (N/mm^2)
Min. values in TS 25	1	4
K5	2.5	10.4
K6	3.0	11.6

Table 8
Material amounts of the mixtures and their alkalinity values

Material amounts					Alkalinity values		
Code	Cement (g)	Cement (%)	Ash (g)	Ash (%)	CaO (m·mol/L)	OH^- (m· mol/L)	pH
A	20	100	0	0	13.0	35	12.40
K5B	18.4	92	1.6	8	9.8	42	12.75
K5C	16.8	84	3.2	16	6.0	54	12.82
K5D	15.2	76	4.8	24	4.8	65	12.95
K6B	18.4	92	1.6	8	9.0	30	12.70
K6C	16.8	84	3.2	16	5.5	35	12.80
K6D	15.2	76	4.8	24	4.4	49	12.93

A, ash ratio 0%; B, ash ratio 8%; C, ash ratio 16%; D, ash ratio 24%.

Ashes so formed do not have binding character, because they are not fine enough to be mixed with cement. Therefore the ashes are ground and screened to sizes between 200 and 90 μm. The specific surface area of the ashes determined according to Blaine air principal is twice the specific surface area of the cement. The physical and chemical properties of the ashes are given in Tables 4 and 5.

2.5. Pozzolanic activity of the ashes

The activities of the pozzolanic ashes were studied using mechanical, chemical, and physical experiments.

2.5.1. Mechanical experiments

Materials were mixed as standard values given in Table 6. Mechanical tests were done according to TS 25 by using 40 × 40 × 160-mm prisms. Mechanical test results are given in Table 7. These results exceed $fb_{min} = 1$ N/mm^2 for bending tests and $fc_{min} = 4$ N/mm^2 compression tests. These observations showed that both ash groups have pozzolanic properties and K6 (ash production at 570°C) has greater flexural and compressive strength than K5 (ash production at 670°C).

2.5.2. Chemical experiments

Pozzolanic activity was studied by chemical experiments according to TS 26. This test is based on the amount of free $Ca(OH)_2$ removed after the hydration of silicates. $Ca(OH)_2$ is released after the hydration of the cement containing C_3S and C_2S and the main parts. When pozzolanic material is

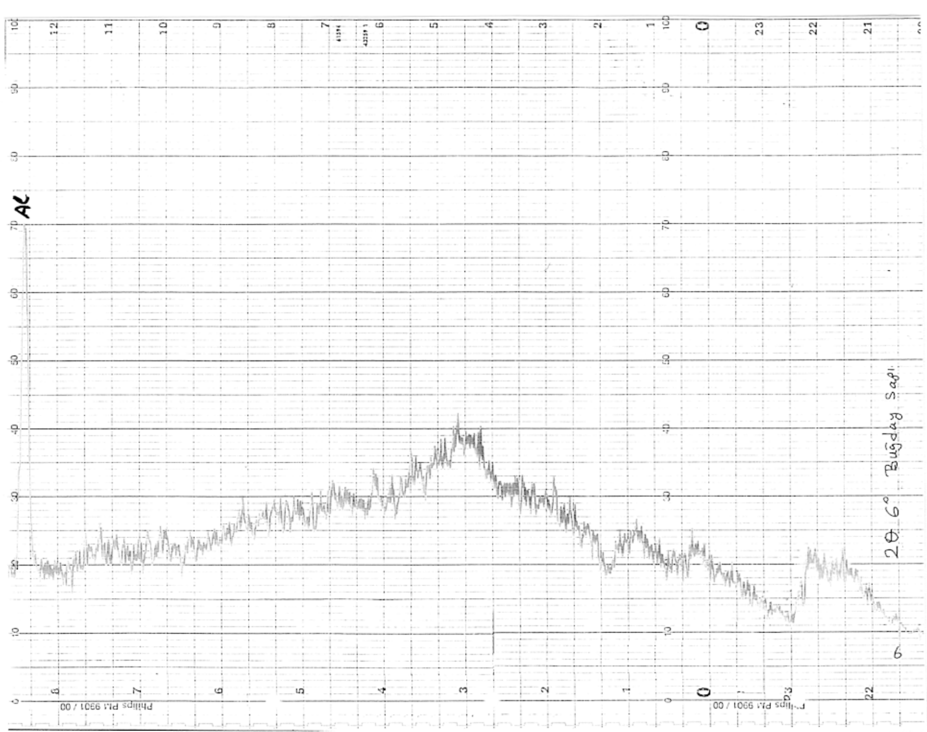

Fig. 1. X-ray diffractogram of wheat straw.

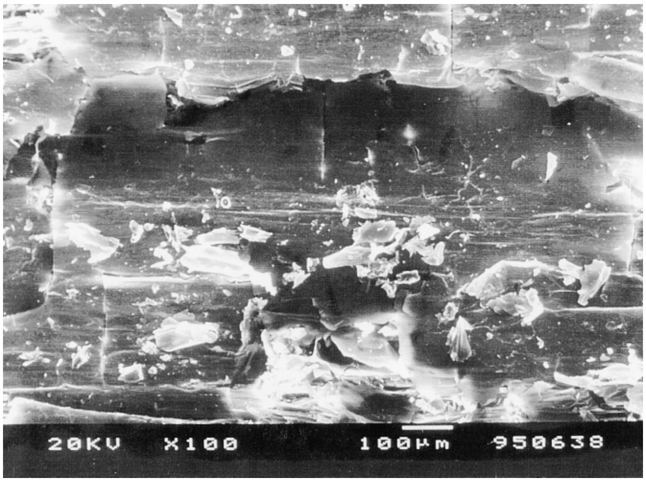

Fig. 2. SEM photograph of wheat straw.

added to the cement, free $Ca(OH)_2$ and active silica can combine and form silicate hydrates, which are not soluble in water. This reduces the free $Ca(OH)_2$ in the system. The reduction of free lime shows that the ashes have pozzolanic activity.

In chemical experiments, ashes were added to the cement in proportions of 0, 8, 16, and 24% and specimens were prepared by mixing materials. The pH values of the specimens filtered were measured and the amounts CaO and OH- were determined. The alkalinity values are given in Table 8. The solubility curve was established according to this value. As can be seen in Fig. 8, ash-added specimens showed alkalinity values below the solubility curve. In both types of ash, the amount of free $Ca(OH)_2$ reduces with increasing ash contents. According to this data, the ashes have pozzolanic characters, since the data points are located on the positive side. K6 group is more active than K5 group.

2.5.3. Physical experiments

Pozzolanic activity was studied by X-ray analysis using a Philips Cu Kα radiation X-ray diffractometer with 2Φ values changing between 6 and 70°. The patterns of the ashes are given in Figs. 4 and 5. K5 and K6 type ashes have no peaks between 13 and 33°, which shows that these ashes are amorphous. This physical state associated to the high SiO_2 + Al_2O_3 content justify the pozzolanic character. Microphotographs of both ashes are taken by SEM and given in Figs. 6 and 7.

3. Conclusions

The following conclusions were determined at the end of the experimental studies.

- Wheat straw has 8.6% ash and the silica content of the ash is 73%.
- Both ashes burned at 570 and 670°C have pozzolanic properties.
- The pozzolanic properties obtained at 670°C are higher than those obtained at 570°C.
- A preburning step reduces the volume of the material, saving a part of the burning energy and preventing the formation of smoke and contaminations.
- Ash obtained from the wheat straw can be used as a pozzolanic materials wherever wheat production is large.

Acknowledgments

The authors would like to thank Yıldız Technical University Research Fund Directorate for supporting this study.

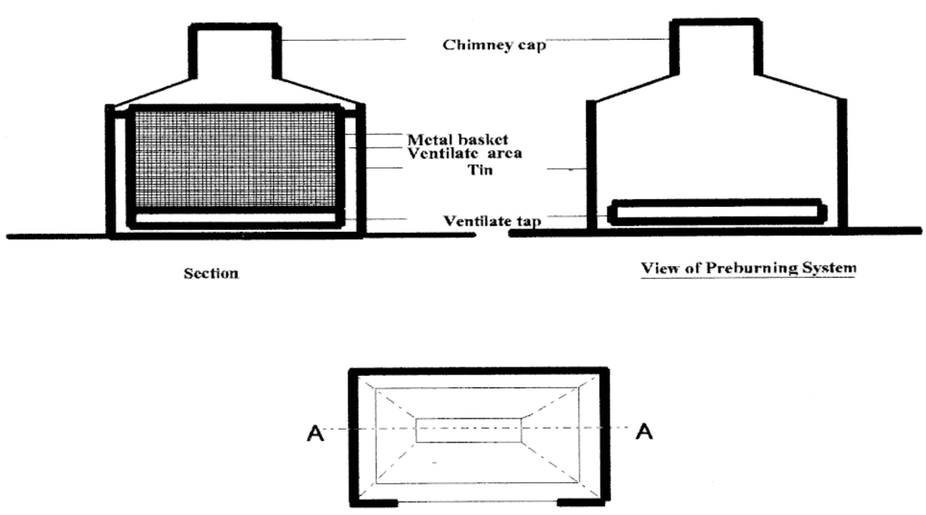

Fig. 3. Preburning system.

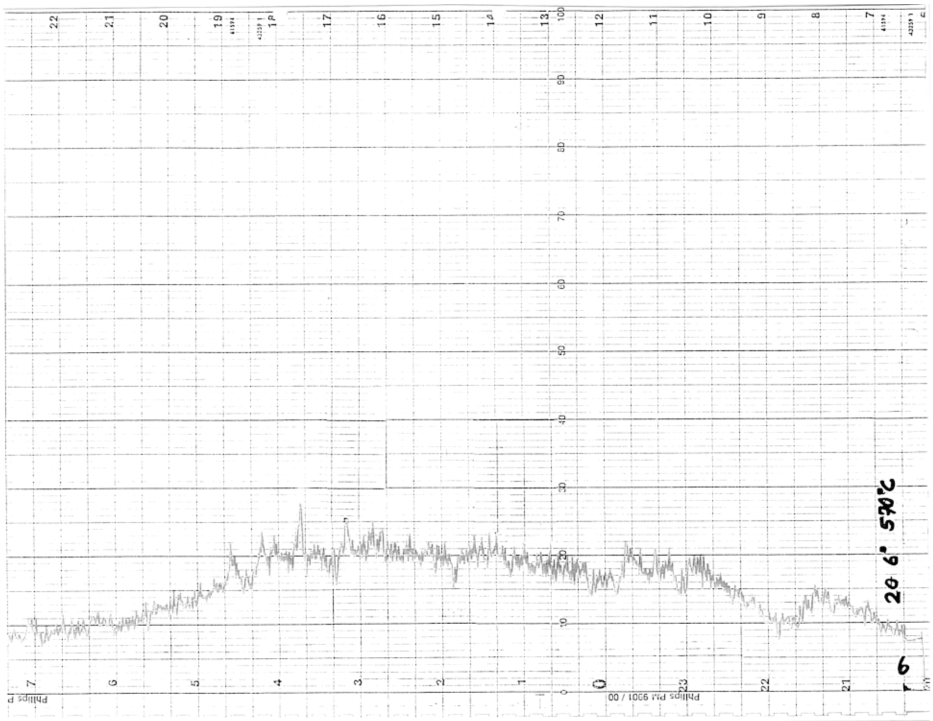

Fig. 4. X-ray diffractogram of wheat straw ash burned at 570°C.

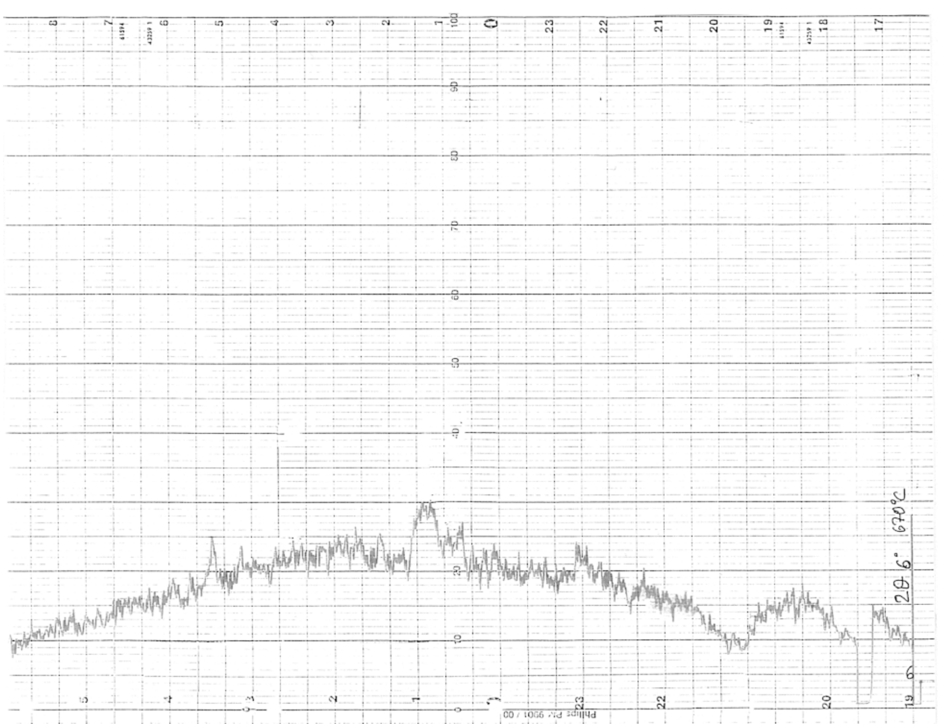

Fig. 5. X-ray diffractogram of wheat straw ash burned at 670°C.

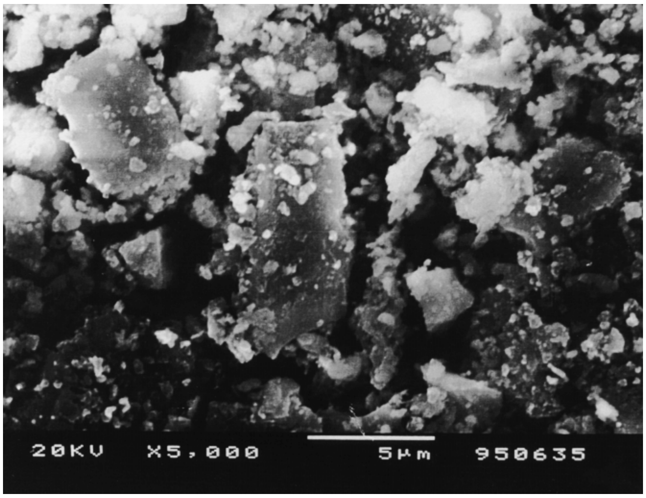

Fig. 6. SEM photograph of wheat straw ash burned at 570°C.

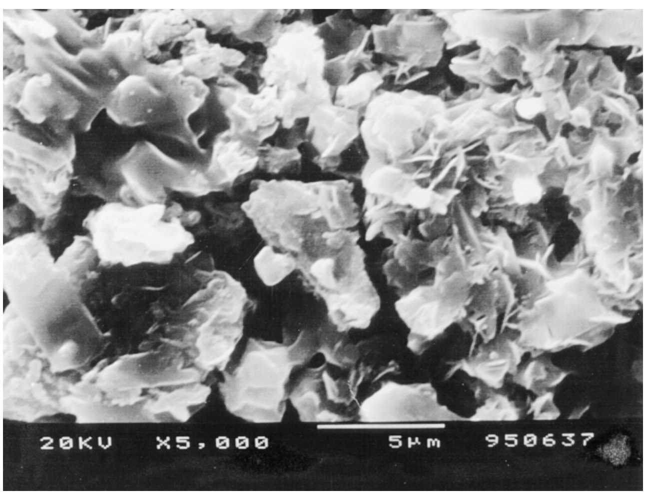

Fig. 7. SEM photograph of wheat straw ash burned at 670°C.

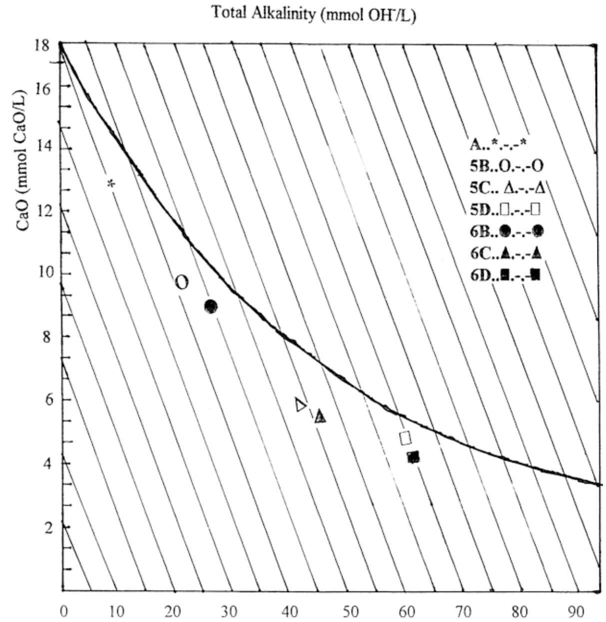

Fig. 8. Ca-OH solubility relationship of ashes to standard values of TS26.

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