Physicochemical properties and silicon content in wheat flour treated with diatomaceous earth and conventionally stored

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ABSTRACT

This study was done to evaluate the physicochemical properties and to quantify the residual silicon in flour from wheat grains treated with different dosages of DE and stored in a conventional system for 180 days. Samples containing 10.0 kg of wheat grain were treated with 0.00, 2.00 and 4.00 g kg^{-1} of DE and then homogenized and stored in cotton bags at 22 °C and 70 ± 5 g 100 g^{-1} relative humidity. Physicochemical analyses were carried out at 0, 60, 120 and 180 days of storage. The experiment was conducted in a randomized design with a factorial 3×4 arrangement (three doses of DE $\times$ four storage periods), totaling 12 treatments, with three replicates for each treatment. The wheat flour samples were digested and the silicon residue quantification was conducted through colorimetry. The wheat treated with DE presented a lower test weight compared with control, $+b^*$ chromaticity coordinate for color and increased ash content and L^*. The physicochemical changes in the grain and wheat flour were proportional to the amount of DE applied.

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1. Introduction

Diatomaceous earth DE is an inert dust, derived from amorphous sediment originating from the carapaces of vegetal unicellular organisms such as aquatic, lacustrine and marine algae. It is a light material with low density and a color ranging from white to dark grey. It consists of approximately 80–93 g silicon dioxide in 100 g and the remaining content is composed by clay minerals, organic matter, hydroxide, quartz, calcium and magnesium carbonate (Stathers et al., 2004; Korunic et al., 1998). Diatomaceous earth can be used for pest control in grain storage (Quarles, 1992). It is both abrasive and slightly sorptive, and dust particles adhere to the insect exoskeleton and remove lipids from the cuticle, causing dessication (Ebeling, 1971; Korunic, 1998). Inert dusts such as DE are most effective when applied as dusts but some retain activity even when applied as a water-based slurry (Golob, 1997). Diatomaceous earth can cause changes to the physical and mechanical properties of grains, increasing the required grinding time for processing and resulting in abrasion of equipment (Miranda et al., 1999).

The classic method for determining the silicon (Si) content in materials is the conversion of insoluble silicate into soluble sodium silicate by fusion of the material under analysis with sodium hydroxide at high temperatures. Silicon content can be determined by colorimetry, gravimetric emission spectrometry and atomic absorption (Sripanyakorn et al., 2005). Colorimetry, based on the formation of a reduced molybdenum-silicon complex or molybdenum blue, is the most commonly used method to determine silicon content. However, the successful use of this method depends on the dissolution of the silicon present in the materials. Several other techniques to measure silicon content exist, including ones in which the sample is fused with alkaline substances such as sodium carbonate and sodium hydroxide, and methods based on fluorization in the presence of excess of boric acid (Elliott and Snyder, 1991; Rodrigues and Santana, 2005).

In the colorimetric method, the reaction product is a blue compound obtained from the heteropolyacid reduction that occurs in two phases: the formation of silicomolybdic acid and reduction by organic or inorganic agents. The method depends on environmental acidity, which can minimize the influence of interfering agents. When the interference is due to phosphates, oxalic acid is often used as a reducing agent (Ferreira et al., 1987; Snyder, 2001).

Although some studies have examined the use of DE in wheat, the literature does not contain information on the amount of residue remaining in the flour after the wheat is ground (Atui et al., 2003). Studies that quantify residual DE in wheat flour are of

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interest to the industry and to official food analysis laboratories because of the potential impact on industrial quality. The objective of this study was to evaluate the physicochemical properties and quantify the residual silicon in flour made from wheat grain treated with different doses of DE and stored in a conventional system for 180 days.

2. Material and methods

The wheat (*Triticum aestivum* L) used in the study was from the 2008/2009 harvest of the cultivar Abalone, produced in an experimental field at Biotrigo Genética Ltd., which is located in Passo Fundo City, Rio Grande do Sul State. The wheat grains were harvested with a thrasher combine, pre-cleaned in an air machine and sieves and dried in a stationary dryer until it reached 13 g 100 g^{-1} humidity. The DE used was KeepDry®, Brazil, with about 93 g 100 g^{-1} amorphous silicon dioxide (SiO$_2$), an average granulometry of 15 μm, a bulk density of 200 g L^{-1} b, a light color, insolubility in water and a dry powder aspect. Water was deionized and all reagents used were analytical quality (Sigma, Aldrich, Carlo Erba and Merck).

The study design was a completely randomized 3 × 4 factorial arrangement (three DE parts × four storage periods) totaling 12 treatments, with three replicates for each treatment. Diatomaceous earth was added to 10.0 kg wheat grain samples in 0.00, 2.00 and 4.00 g kg^{-1} dosages. Samples were homogenized in a Hypo planetary mixer (Model HB 25) and stored in cotton bags at 22 °C and 70 ± 5 g 100 g^{-1} relative humidity. Physicochemical analyses were conducted at 0, 60, 120 and 180 days of storage. The wheat was ground in a Chopin pilot mill (Model CD1, France) according to the n. 26-10 AACC method (2000). The analyses were performed for 0.100 g samples, with two replications.

The test weight was determined according to the Seed Analysis Regulation (Brazil, 1992), and results were presented in kg hL^{-1}. The ash content of the wheat grains was determined in accordance with the n. 08-01 AACC method (2000). Analyses were carried out in duplicate and results were presented in g 100 g^{-1}. The color of the wheat flour samples was determined in triplicate using a Hunter-Lab® spectrophotometer (ColorQuest II, England) with a spherical geometrical optical sensor according to the n. 14-22 AACC model (2000). The intensity values of the L^* component (brightness) and chromaticity coordinate $+b^*$ (yellowness) were also determined.

The wheat flour samples were digested according to the procedure proposed by Elliott and Snyder (1991), with changes in time and incineration temperature and in the amount of NaOH solution. The samples were weighed, transferred to nickel crucibles and heated at 650 °C for 4 h. The residue was cooled, and then 3.7 mL of 12.50 M NaOH solution was added. The crucibles were heated slowly to allow the solvent to evaporate. The solids were redissolved in 35 mL deionized water, and the resulting solution was left to sit for 24 h. The crucibles were then washed with 15.0 mL of 0.100 M NaOH solution, transferred to a polyethylene bottle and the volume completed to 50 mL with deionized water. To assess the applicability of this method, known amounts of DE were added to 0.00−0.100 g samples of wheat flour, which were digested as previously described. Additionally, 0.100 g DE samples, wheat flour free, were similarly digested and used to obtain the analytical curve. The experiments were performed with two replications.

Silicon determination was conducted with a Shimadzu UV−Vis spectrophotometer, (Model 1800, Japan), which operates at wavelengths from 200 to 1100 nm. Silicon content was determined according to the method described by Furlani and Gallo (1978), with changes in the proportion of the reagents used. The spectrophotometer was calibrated with deionized water containing all the reagents except for the sample. For the analyses, 5.0 mL volumetric balloons with 10 μL of the digested sample and 10 μL of 2.50 M sulfuric acid solution, which was enough to stabilize the pH between 4.5 and 5.0, were used. Two hundred microliters of 0.043 M ammonium molybdate were added to the volumetric balloon, and the solution was shaken and allowed to sit for 5 min. Next, a newly prepared solution containing 100 μL of 1.10 M oxalic acid, 30 μL of 2.50 M sulfuric acid and 100 μL of 0.110 mol M ascorbic acid was added. The volume was completed with deionized water and the solution was homogenized and allowed to sit for 15 min. The absorbance reading was conducted at a wavelength of 815 nm. For the silicon content analysis, 1000 μL samples were used, and the remaining reagents were present in proportional amounts. The standard curve for silicon determination was built with the DE solution, treated as previously described, in a concentration range of 0.0 to 7.37×10^{-5} M.

ANOVA was performed on all physicochemical determinations results using a statistical program Sisvar® Versão 5.3, Build 75 (Ferreira et al., 2010). Analyses and regression equation graphs were elaborated with the support of the Origin 5.0 program.

3. Results and discussion

The variance analysis mean squares for storage time, DE dosages and interaction time × dosage were significant ($P \leq 0.05$) for the L^* component of intensity (brightness); the $+b^*$ component of color chromaticity (yellow); and for the ash content and test weight of wheat grains treated with DE and stored for 180 days (Table 1). The variation coefficients revealed values with reduced magnitude, reflecting suitable control of these techniques during the experiment as well as the accuracy and reliability of the study outcomes.

The flour color was evaluated by measuring brightness and yellowness. Brightness is affected by the presence of meal or foreign material, whereas yellowness is related to the number of pigments present. The flour's color is defined with the tridimensional color scale, which describes the different color components. Reflected light is provided by a dark or bright component in addition to a red or green and a blue or yellow component, as determined by calorimeters or spectrophotometers (Peterson et al., 2001).

After storage for 180 days, the wheat flour became lighter and the L^* (brightness) component increased significantly; the regression equations DE0, DE20 and DE40 presented determination coefficients between 0.89 and 0.97 (Fig. 1A). The DE0 samples presented higher L^* (brightness) color component intensities at the beginning and maintained the highest brightness levels throughout the entire storage period compared with the other samples. On the other hand, the wheat flour treated with 4.00 g kg^{-1} of DE presented lower values of L^*.

Table 1
The variance analysis results for L^* components (brightness), flour color $+b^*$ (yellow) and the ash content and test weight (TW) of wheat grains treated with DE and stored for 180 days.

Variation[a]	Mean squares				
Source	DF	L^*	$+b^*$	Ash	TW
Time (days)	3	2.32[a]	1.62[a]	0.016[a]	4.05[a]
Dosage (g)	2	9.36[a]	6.43[a]	0.139[a]	150.22[a]
Time × dosage	6	0.09[a]	0.019[a]	0.001[a]	0.32[a]
Total	35			23	35
VC		0.0006	0.0041	0.0087	0.0026

DF − Degrees of freedom; VC − Variation coefficient.
[a] Significant at the level of 0.05 probability ($P \leq 0.05$).