



Effect of sodium compounds on the sintering propensity of coal-associated minerals



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ARTICLE INFO

Article history:

Received 30 April 2014

Accepted 7 December 2014

Available online 9 December 2014

Keywords:

Sintering

Minerals

Compressive strength

NaCl

Na₂CO₃

ABSTRACT

This study focused on the chemistry involved in sintering of coal-associated minerals during combustion and inert conditions. Changes in mechanical strength were recorded with compressive strength measurements. This method was used to investigate the influence of sodium addition (as NaCl and Na₂CO₃) on the sintering strength of a prepared mineral mixture at temperatures ranging from 500 °C to 1000 °C. Supporting data were collected with thermogravimetry (TG), differential thermal analysis (DTA), scanning electron microscopy (SEM) and X-ray diffraction (XRD). It was found that the addition of both NaCl and Na₂CO₃ increased the sintering of the mixture by between 20% and 80% at temperatures ranging from 500 °C to 800 °C in air. The increase in sintering was most likely due to the enhanced sulphation of limestone to anhydrite, resulting in a more extensive sintered network. Anhydrite decomposes at temperatures higher than 900 °C. At 900 °C and 1000 °C the sintering observed for the mixture with additives was comparable to the sintering observed for the baseline mixture. The decomposition of anhydrite therefore weakened the sintered network to the same level as the baseline mixture. In an inert atmosphere (N₂) the same strength development as in the mixture heat-treated in air was not observed in mixtures with sodium additives. Anhydrite was not formed in N₂ due to the lack of O₂ and SO_x. A decrease in the sintering propensity upon heat-treating the samples from 700 °C to 1000 °C in N₂ for the experiments containing NaCl as additive was noted. This was most likely due to the volatilization of NaCl that weakened the structure. The addition of Na₂CO₃ increased the sintering propensity of the mixture heat-treated in N₂ from 500 °C to 800 °C. The mechanism for this increase is currently unknown and should be further investigated.

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1. Introduction

Sintering is relevant to many technologies, including coal processing. Sintering and agglomeration are associated with particle growth and can cause unstable or problematic operation during thermal processing of coal, especially in fluidized bed reactors. If the sintered particles become too large, partial or complete defluidization of the bed will occur. Sintering and agglomeration are more problematic in the utilization of low-rank coals due to the presence of significantly larger amounts of organically bound inorganic matter that are released at low temperatures [1–3].

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Of particular concern in low-rank coals are high amounts of alkali metals (e.g. sodium and potassium) [2–5]. Two forms of sodium that are associated with coal utilization are NaCl and Na₂CO₃. Kyi and Chadwick [6] reported that NaCl is the primary sodium species released in the gaseous phase from coal during combustion. High concentrations of NaCl are found in “saline” coals, e.g. some coals from the UK, Russia, Ukraine and USA [7]. Kosminski and co-workers [8] reported that Na₂CO₃ formed as the principal form of sodium from carboxylate sodium during pyrolysis and gasification of coal.

Many studies have focused on the role of sodium in the defluidization of beds and the presence of sodium in the molecular structures of the particle bridges [1–3,9–11]. Bhattacharya and Harttig [1] and Manzoori and Agarwal [2,9] studied agglomeration and defluidization of high alkali and high sulfur low-rank coals during fluidized-bed combustion. They found that the agglomeration in

the systems was mostly caused by alkali, magnesium and calcium sulfates. Eutectics can form in the ternary systems with melting points in the range of 650–940 °C [9,10].

This study focused on the chemistry involved during sintering of the minerals associated with coal under both oxidizing and inert conditions in the temperature range of 500–1000 °C. The sintering of ash in combustion systems is a well-known phenomenon and the subject of an extensive literature [12–15]. Coal ashes share the common property with many (though not all) ceramic materials that the compositions of both ashes and traditional ceramics are dominated by silicates and aluminosilicates. Articles or pieces of silicate-based ceramics are formed by vitreous sintering. Therefore, it is reasonable to expect a similar mechanism, with the production of an intermediate liquid phase, to be responsible for sintering in coal ashes. Raask [13] has discussed this topic at length. The fundamental physics and chemistry of sintering phenomena in general are covered in the monograph by German [16]. With respect to the temperature range used in the present work, and the possible relevance to fluidized-bed combustion, it is known that sodium is the single most important factor in determining severity of bed agglomeration, and these problems can be expected as the sodium concentration in the bed approaches 4% [17]. Similar observations have been reported for Turkish lignites [18].

An increase in mechanical strength of the agglomerates formed during coal combustion is associated with an increase in sintering [19]. Changes in mechanical strength were recorded with compressive strength measurements. The method was used to investigate the influence of sodium addition (as NaCl and Na₂CO₃) on the sintering strength of a prepared mineral mixture comprising of kaolinite (Al₂Si₂O₅(OH)₄), quartz (SiO₂), pyrite (FeS₂), calcite (CaCO₃), FeCO₃, MgCO₃ and TiO₂. The major chemical changes associated with heating the minerals in this mixture, as well as the compression strength and temperature at the onset of sintering are presented elsewhere [20,21]. In short, investigation into the characteristics of the mineral mixture heated in air indicated that sulphation of limestone (formation of anhydrite) is the major contributor to strength development in the mineral mixture pellets [20,21]. Supporting data for the influence of sodium compounds on the sinter strength of this mineral mixture were collected using thermogravimetry (TG), differential thermal analysis (DTA), scanning electron microscopy (SEM) and X-ray diffraction (XRD). Insights gained can be applied in coal combustion processes to assess the influence of sodium on the formation of sintered material, as well as predicting the influence on the strength of this material.

2. Materials and methods

2.1. Mineral mixture and additives

The mineralogy of different types of coal varies greatly. In order to eliminate the influence of minor and trace species already present in coal, an arbitrary mineral mixture was prepared consisting of the major minerals generally present in coal. We recognize that an alternative approach would be to select one coal as a reference coal, and then to dope its ash with the sodium compounds. However, given the great variability of mineral assemblages and ash compositions in the world's coals, and given the potential for unknown effects caused by the minor and trace elements already in the coal, we chose to create a model mineral mixture to be doped with the sodium compounds. With respect to selection of components for the baseline mixture, we chose representatives of the major mineral families known to occur in most coals, i.e., clays, carbonates, sulphides, quartz, and oxides or hydroxides [22–25]. Kaolinite is a dominant member of the clays found in coals; calcium,

magnesium, and iron are the important carbonates; and pyrite is the most important sulphide [13,22–26].

The mineral mixture was prepared consisting of 25% kaolinite (Al₂Si₂O₅(OH)₄), 20% quartz (SiO₂), 20% pyrite (FeS₂), 15% calcite (CaCO₃), 8% FeCO₃, 8% hydromagnesite (MgCO₃, basic), and 4% anatase (TiO₂) by weight percentages, as previously reported [20,21,27]. The weight percentages of the different components were selected based on their relative abundances in coal, but high enough to be quantified with the analytical techniques utilized in this study. The compounds (as received) were mixed in the appropriate ratios and dried overnight at 100 °C. The mixture was sized to –355 μm. Sodium chloride (NaCl) and sodium carbonate (Na₂CO₃) were added to the mineral mixture as additives at a concentration of 4% by weight (1.6 g and 1.7 g Na, respectively).

2.2. Compressive strength tests

Cylindrical pellets were prepared in a 10 mm die set from 1 g of the mineral mixture. The method used to prepare the pellets, as well as the heat-treatment and crushing methods employed have been described elsewhere [20,21]. In short, the green pellets were cured in oxidizing (air) and inert (N₂) conditions to temperatures between 500 °C and 1000 °C, in 100 °C increments. The cured pellets were crushed under ambient conditions using a strength tester to record their compressive strengths. Each batch of pellets cured in the furnaces also contained mineral mixture pellets (without additives) to serve as baseline references. The average compression strength values were calculated for each experiment. The values are available in the supplementary data. The average values obtained for the mineral mixture experiments (without sodium-containing additives) were subtracted from the values for the relevant experiments containing sodium additives. The focus of the study was not on the sintering of individual components in the mineral mixture. Subtracting the values of the mineral mixture experiments from the values obtained for the mineral mixture doped with the sodium compounds ensured that only the added effects from the sodium compounds were studied. The differences are expressed as a percentage (deviation) from the average values obtained for the mineral mixture pellets (baseline). The formula is given below.

$$\% \text{Deviation} = \left(\frac{\text{Average}_{\text{Additive}} - \text{Average}_{\text{Mixture}}}{\text{Average}_{\text{Mixture}}} \right) \times 100 \quad (1)$$

The graphs depicting the compressive strength results obtained for the experiments containing additives show the mineral mixture (no additives) results as the baseline reference or zero line, as well as the percentage deviations from the zero line for the experiments containing additives. The error bars associated with each average value represent 95% confidence limits.

2.3. Simultaneous thermogravimetry and differential thermal analysis (TG/DTA)

Mass losses of the mineral mixture (with and without additives) were measured with a Mettler Toledo TGA/SDTA 851° system. Approximately 20–80 mg of sample was heated from 30 °C to 1000 °C at 5 °C/min in a gas flow of 150 mL/min of either air or nitrogen. The DTA curve for each sample was determined simultaneously.

2.4. Scanning electron microscopy (SEM)

SEM analyses were performed on sintered pellets with a FEA Quanta 200 ESEM instrument equipped with an Oxford INCA X-Sight 400 EDS (energy-dispersive spectrometry) system and operated with a 15 kV electron beam. Backscattered electrons combined with EDS were used to determine the elemental composition

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