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Methodology of sample selection for study of limestone decarbonation

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Abstract

Depending on nature of calcination process, we expect to get lime with specific features, for example fast or slow reactivity, certain grain size or lime that provides hydrated lime with high specific surface area. However, reality is often completely different. Because of that, there are dozens of works in past 70 years that tried to find correlation between properties of limestone, calcination parameters and final properties of hydrated lime. None of these papers has managed to find such a relation in general sense. Our work on this subject starts with proper sample selection. In order to contain the widest range of samples, we selected only limestones with high purity. All the samples have content of CaCO₃ over 97.8 %. Sample file contains specimens with various geological age (from 60 mil. to 500 mil. years old), various physico-mechanical properties and different petrology. These samples are complemented with some limestones with unexpected behavior during calcination process. Rationale behind selection of each sample is included. We ended up with 26 samples of limestones. These samples were in first phase analyzed using chemical analysis, mineralogical analysis of insoluble residues and many others to identify differences in behavior. The results showed differences in the porosity and the associated volumetric mass density. Mineralogy of insoluble residue was in all cases very similar, so it is not cause of difference in behavior. This means that there are many other factors left for assessment of effect on behavior of lime, hydrated lime and also limestones during burning process.

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1. Introduction

Lime is one of the oldest building materials known, dating back over 3000 years ago. Historically, there were not very high demands on its properties. Growth of industrial production, particularly steel production and later growth of industrial chemistry and modern building material began to emerge higher requirements on specific properties of lime, particularly with regards to its reactivity. Similarly the requirements for strength of lump lime became higher to reduce decrepitating and thereby reduce dustiness during manipulation. However it appeared that not all limestones can produce lime with required properties. Some limestones, even those with high purity can quickly become hard burnt, others are unable of hard burn despite of excess of burning energy. Some limestones fall apart during burning, other are willing to sinter. Similar variance is in properties of burnt lime. This issue is significant and it bothers both manufacturers and users. Because of it, there were a lot of studies and publications within past 50 years in field of limestone, their decarbonation and properties.

Wuhrer (1953, 1956) studied problems of decomposition of limestones during burning process [1,2]. He was first to describe penetration lines of dissociation in a thin layer from surface to the center of the sample. He also studied size of pores and their total volume on various limestones, but failed to discover clear dependence to process of dissociation. Penetration rate of dissociation lines was described by Hedin (1956) [3]. His study shows that dissociation rate is controlled by escape speed of CO₂ to surface. This effect was described in more detail by Cremer and Nitsch in 1959 [4]. Following these works, Kainer et al. (1982, 1986) attempted to determine coefficient rate of dissociation reaction base on diffusion coefficients relative to porosity of limestone [5,6].

Great attention was also given to disintegration of limestones during firing process, because it is significant technological problem when using shaft furnace. This topic was summarized by Schiele and Behrens (1972), who found correlation between crystal size and disintegration of limestone [7]. Extreme cases of disintegration are seen when firing marbles, which are perfectly crystalline.

There was effort to describe sintering process in the end of 19th century, with no result. Clark (1940), Wuhrer (1953) and Pohl (1963) were first to start systematic study of growth of CaO crystals with increasing temperature and correlation with density and reactivity of lime [8,9,1,10]. Sintering process can take place even when there is still unfinished decarbonation inside of sample, as was demonstrated by Pohl (1956) [11]. He also confirmed that when sintering occurs, density of sample increases with temperature. Temperature of core of samples, which were not decomposed, was in all cases lower than the decomposed limestone. Lehmann and Schmidt (1961) recount shrinkage of samples depending on the sintering process [12]. They also distinguish decarbonated lime and sintered lime, but it is very difficult to separate one from another, because both processes take place almost simultaneously.

Hogewonning and Wolter (2004) summarize in their work all of the existing knowledge of most important historical studies in this field [13]. They also made complex study of limestones from all 23 existing lime plants in Germany themselves. They used latest analysis technologies and focused on petrographic and physico-chemical properties. They exposed their samples to various temperatures and monitored various parameters of limes and limestones with effort to create mathematical models. These are the most significant findings:

- Sintering process was observed in all samples from 1050°C with one hour of endurance and it is dependent on specific surface of limestone
- Limestones with larger specific surface area have, because of larger reaction area and therefore lower diffusion coefficient, higher potential for hard burning.
- Limestones can be, based on characteristics of produced limes, divided into three group.
 1. Limestones with higher percentage of impurities and thus lower content of active CaO. This group exhibits certain stopping of sintering process. Impurities inhibit diffusion process of solids at the interface between individual crystallites of CaO.
 2. Limestones with moderate amount of impurities (especially K₂O and MgO) create mainly limes with small grain size and with crystal structure of CaO penetrated with impurities.
 3. Third group consists from highly pure limestones. In these, diffusion of solid particles is not obstructed and because of that is sintering process very fast. These limestones have higher prerequisite to create hard burnt lime.

Despite all the work performed, we couldn't find sufficient answer why some limestones behave differently than

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