Summary

Phase condition of crushed concrete sand

Crushed concrete sand from recycling plants contains a mixture of mineral components. Its ratio of hardened cement paste to aggregate is much higher than the ratio of the originally used concrete. Crushed sand consists of great amounts of hydrated cement phases as well as amounts of calcium hydroxide (portlandite, $Ca(OH)_2$) and calcium carbonate (calcite, $CaCO_3$). The aggregate of concrete sand mostly consists of quartzitic and/or carbonatic phases; moreover, there are several additives often used for concrete such as trass, fly ash, blast furnace slag, silica fume and others.

The main phases of hardened cement paste are:

- calcium silicate hydrate (50-60%)
- calcium hydroxide (20-25%)
- hydrated aluminate-, ferrite-, and sulfate phases (10-15%)
- traces of non-hydrated cement

Phase reactions of concrete at elevated temperatures

During drying processes free water and starting at temperatures of about 100°C parts of physically bonded water are set free. These processes remain nearly constant in the temperature range between 100°C and 200°C. At temperatures below 300°C chemically bonded water (dehydration) is already released, combining the processes of decomposition, transformation and new growth of phases. These processes and reactions cannot be related to an exactly defined range of temperatures as the cement stone, which consists of many compounds, as composition and hydration conditions. The dehydration of hydrate phases carries on continually at higher temperatures, but complete dehydration of the CSH-phases needs temperatures highly above 300°C. Between 450 - 550°C the decomposition of calcium hydroxide into calcium oxide and water starts. At 573°C a transformation of quartz happens. This so-called quartz inversion is a structural transformation from α-SiO_2 to β-SiO_2, generally also known as transformation from low- to high quartz.

For a thermal treatment of crushed concrete sand, transformation and decomposition of hydrate phases starting at 400 - 600°C are the most important reactions. With the decomposition of CSH-phases the simultaneous forming of C_2S-phases occurs and thereby hydraulically reactive parts are formed into crushed concrete sand. The formation of reactive C_2S is very important for the thermal conditioning of crushed concrete sand. The controlling temperature range for the new growth of the C_2S phase is between 650-900°C.

Another important reaction for the thermal treatment of crushed concrete sand is the decarbonization of calcium carbonate, which results in an increase of free lime (CaO). The latter is also formed by the following reactions: by decomposition of portlandite and calcite and through degradative reaction of CSH-phases at high temperatures. Hence, a part of free lime is integrated in the newly grown phases during the decomposition process of the CSH-phases and the formation of C_2S. Due to that fact the content of free lime increases continually with rising temperatures and temperatures above 800-900°C.

The effort to treat crushed concrete sand at very high temperatures to get hydraulic products must guarantee that the content of free lime is very low to avoid expansion of concrete or mortar which has been produced with thermally treated crushed concrete sand.

Investigations of production of hydraulic active phases

Tasks

The aim is to give or give back hydraulic properties to crushed concrete sand which makes it possible that this material gets a new use as a binder component for the production of mortar or concrete.

Materials and the program of tests

The crushed concrete sand was produced from concrete. Cement, type CEM I 32,5 R, acc. to DIN 1164 was used as binder. The water-cement-ratio was 0,5 and quartzitic gravel within the grading curve range of A/B 16 acc. to DIN 1045 was used as aggregate. The production was done in cylindrical forms. The samples were stored for 7 days in water before they hardened in a climatic chamber at 20°C and 65% relative humidity.

After a period of ≥ 180 days the samples were crushed with a jaw crusher and the crushed concrete sand was sieved to < 0,25 mm. The determination of binder content in these fine particles of the crushed concrete sand acc. to DIN 52170 showed a content of 50 % binder and 50 % aggregate.

The fine crushed concrete sand (< 0,25mm) was treated with temperatures between 400°-1000°C at intervals of 50°C. The cooling down process was the same for all samples, spreading on a sheet at room temperature. An x-ray phase analysis was done before and after the thermal treatment (chapter 6). With tempered crushed sand as binder and standard sand as aggregate mortar prisms were produced acc. to DIN 18555, part 1, with a binder to aggregate ratio of 1:3 by volume parts. The results shown in the following give an average value for a series of measurements on three mortar prisms. The mortar prisms were stored at 20°C/65% relative humidity or 20°C/under water. Mechanical properties of the prisms were determined after 7 and after 28 days (chapter 7).

Test results

Thermogravimetric analysis

From the TG/DTG and DTA curves in chapter 6 the phase changes temperatures below 1500°C can be seen.

Up to about 105°C drying takes place. At about 450°C there is degradation of portlandite and CSH-phases and between 750-800°C calcium carbonate is decomposed. As a result, 700°C was chosen as a suitable temperature of treatment. For this temperature a relatively high C_2S-content can be expected together with a low content of free lime.

Phase analysis by x-ray diffraction method

The formation of C_2S after a treatment with temperatures of 700°C can be analyzed with x-ray diffraction (x-rays used: $CuK_{\alpha 1}$). In chapter 6 the intensities of untreated crushed concrete sand (lower curve) and those of crushed concrete sand after temperature treatment are shown. Before treatment the reflexes of CSH-phases, after treatment the presence of C_2S are marked. In both diagrams the reflexes of quarzitic aggregate (quartz) within crushed concrete sand are marked.

Determination of CSH-phases with SEM

The newly grown CSH-phases, causing the development of mechanical strength, can be proved with the help of a scanning electron microscope (SEM) (see chapter 6).

Variation of temperature level

In order to confirm the optimal treatment temperature of 700°C for the crushed concrete sand examined here, compressive strength was determined for mortar prisms acc. to DIN 1164 for a temperature range between 400-1000°C.

The results given in chapter 7 show that the treatment with a temperature of 700°C results in crushed concrete sands with highest levels of strength. Below 700°C there is not enough C_2S for hydraulic reactions. For higher temperatures a loss of strength is observed due to a growing amount of free lime (CaO) which has a weakening effect.

Variation of maximum grain size

For ecological and economical aspects it is important to choose a maximum grain size of crushed concrete sand as large as possible. Therefore crushed concrete sands with different maximum grain sizes were treated thermally and used as binder in mortars afterwards. Chapter 7 shows that the highest compressive strength of mortar can be reached if the thermally treated crushed concrete sand had a maximum grain size up to 0,25 mm. A finer grinding or sieving does not result in higher degrees of strength. An increased maximum grain size results in a drop of strength, because the reactive surface is smaller.

Variation of holding time

After determining the optimal temperature of treatment, the duration of treatment was optimized. Chapter 7 shows that the highest degrees of strength can be reached by a holding time of 30 minutes. Shortening the period of treatment does not result in the phase changes wanted. A period of treatment longer than 30 minutes is more negative, because of growing production of free lime.

Variation of storage of the mortar prisms

Comparing the degrees of compressive strength after 7 or 28 days it can be seen that depending on the way of storing there is an increase of strength for storage under water and a loss of strength for storage in climate 20/65 after the 7th day, related to the 7-days- strength. The cause of a loss of strength for storage 20/65 is a stop of hydration due to drying.

Résumé

By means of thermal conditioning of crushed concrete sand hydraulically reactive C_2S-phases can be produced in the sand. As identified, hydraulic properties are gained back for crushed concrete sand, which make it possible to use it as a binder for the production of mortars. 700°C is the optimal temperature for treatment if C_2S is supposed to be produced in crushed concrete sand. The optimal holding time in a laboratory oven or the holding time in a rotary kiln is 30 minutes. Mortars produced from thermally conditioned crushed concrete sand as a binder have a higher porosity than mortars that were produced with usual cement. The porosity can be reduced by careful follow-up storage, which also results in higher degrees of strength. Mortars belonging to mortar group IIa ($f_C > 7$ N/mm²) acc. to DIN 18555 can be produced without using ordinary cement. In concrete it is possible to substitute the ordinary cement content up to 50% by thermal treated crushed concrete sand. Further it is possible to produce a kind of "lime-sand-stones" from crushed concrete sand after an extra autoclaving process (chapter 8).