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An Alkali Activated Binder for High Chemical Resistant Self-Leveling Mortar

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Abstract

This paper reports the development of an Alkali Activated Binder (AAB) with an emphasis on the performance and the durability of the AAB-matrix. For the development of the matrix, the reactive components granulated slag and coal fly ash were used, which were alkali activated with a mixture of sodium hydroxide (2 - 10 mol/l) and aqueous sodium silicate solution (SiO₂/Na₂O molar ratio: 2.1) at ambient temperature. A sodium hydroxide concentration of 5.5 mol/l revealed the best compromise between setting time and mechanical strengths of the AAB. With this sodium hydroxide concentration, the compressive and the 3-point bending tensile strengths of the hardened AAB were 53.4 and 5.5 MPa respectively after 14 days. As a result of the investigation of the acid resistance, the AAB-matrix showed a very high acid resistance in comparison to ordinary Portland cement concrete. In addition, the AAB had a high frost resistance, which had been validated by the capillary suction, internal damage and freeze thaw test with a relative dynamic E-Modulus of 93% and a total amount of scaled material of 30 g/m² after 28 freeze-thaw cycles (exposure class: XF3).

Keywords

Alkali Activated Binder, Geopolymer, Durability, Chemical Resistance

1. Introduction

The increased demand for the development of alternative building materials has been encouraged by the growth of the building industry and the increased performance requirements together with the higher sustainability criteria applied in civil engineering. Alkali Activated Binders (AAB) represent an attractive alternative for the partial or complete substitution of Portland cement in the production of concrete and mortar. The potential benefit from the replacement of Portland cement by AAB is the reduction

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in carbon dioxide emissions. The production of 1 ton of Portland cement releases approximately 1 ton of carbon dioxide and requires about 100 kWh [1] [2]. As opposed to this, the production of 1 ton of AAB creates only between 0.2 and 0.5 ton of carbon dioxide [3]-[5]. In comparison with composite cements or slag cements, the AAB is the better ecological solution as long as the amount of Portland cement is more than 50% by mass. Thereby, they offer more green benefits than commonly used

CEM II shows a minimum Portland cement portion of 65% by mass. Beyond this, AAB points up a high mechanical strength, a high temperature and fire resistance, an acid resistance and a low shrinkage [6]-[8]. Various scientific reports concerning the microstructure of different AAB are available [9] [10]. Several current studies deal with the production of AAB and their durability properties [11] [12]. The durability is a very controversial property of AAB. Some authors state it as the most important advantage over Portland cement, while others consider that as an unverified statement [13]-[15]. Similar to concrete, where a combination of mechanical and environmental loading can lead to the deterioration of mechanical properties, the durability of AAB is limited by material properties like water permeability, porosity, sorptivity, sulphate resistance, chloride ion penetration, carbonation, corrosion resistance, drying shrinkage and acid resistance [13] [15].

Actually, in comparison to Portland cement, the results of the latest research demonstrate better mechanical properties and durability under aggressive conditions for numerous reaction products made of AAB [16]-[21]. Furthermore, the investigations of Weil *et al.* show an outstanding durability for selective optimized AAB [22]. Malone already showed an enhanced frost resistance of AAB due to a comparatively high density and reduced porosity [23]. However, a noteworthy point is the formation of efflorescence products when AAB products are in contact with water or humidity [23]-[26]. The elution of the alkalis and their reaction with atmospheric carbon dioxide can lead to the formation of carbonates which appear as efflorescence on the surface [24] [25] [27]. An improvement of the durability can be implemented by a high amount of amorphous zeolite phases in the product realized by an adapted silicate modulus [28].

Alkali activated binders are prepared by the alkaline activation of aluminosilicate materials such as fly ash, granulated slag or calcined clays [7] [29], and cured at ambient or slightly higher temperature [30] [31]. The alkaline activation, which comprises the solving process, polymerization and crystallization, depends on many parameters, including chemical composition, physical properties (e.g. particle size distribution and specific surface area of the raw materials), composition of alkaline solution and curing temperature [32] [33]. According to the law of mass action, the amount of amorphous phases formed by the reaction increases with increasing concentration of the alkaline solution [34]. Therefore, strength and durability increase, too [35]. For a further enhancement of mechanical properties of the AAB, short fibres can be integrated [36] [37].

This paper presents results of the development of an AAB-matrix with an emphasis on the measurement of the performance and durability of AAB-matrix.

2. Materials and Methods

2.1. Components for Alkali Activated Binders

In this study, the alkali activated binders were compared with an ordinary Portland cement concrete (OPCC). The compositions of the alkali activated binders are present in **Table 1**. For the reactive binders, the coal fly ash (bulk density: 2.42 g/cm³) "EFA-Füller" (class F) by the company Bauminerale GmbH and a granulated slag (bulk density: 2.85 g/cm³) by the company Holcim were used. The oxides compositions of the coal fly ashes and slag were determined by energy dispersive X-ray spectroscopy (EDX) and are shown in **Table 2**. The measured Blaine fineness of the coal fly ash and of the granulated slag was 7290 and 3740 cm²/g respectively. A quartz sand with a grain size of 0.1 to 1.0 mm was used as aggregate. The alkali resistant (AR) glass fibres, which lower the propensity to shrinkage cracking and can enhance first crack tensile strength in the concrete, were 12 mm long and had a length mass of 45 tex. The activator solutions consisted of a mixture of aqueous sodium silicate (SiO₂/Na₂O molar ratio: 2.1) and a solution of sodium hydroxide.

The sodium hydroxide solution was prepared by dissolving sodium hydroxide pellets (99% purity) in distilled water to a concentration of 2 - 10 mol/l and cooled to room temperature for 24 h. The water/binder ratio was 0.41.

Table 1. Composition of AAB and OPCC in wt%.

	AAB	OPCC
Granulated slag	22.5	-
Coal fly ash	9.0	-
CEM I 52.5	-	22.7
Quartz sand 0-2 mm	49.3	68.9
NaOH-solution	13.7	-
Sodium silicate	4.5	-
AR-glass fibres	0.2	
Superplasticizer	-	0.6
w/b	0.41	

Table 2. Oxides compositions of coal fly ash and granulated slag in wt%.

	Coal fly ash	Granulated slag
SiO ₂	45	52
Al_2O_3	10	30
Fe_2O_3	-	10
CaO	35	5
MgO	10	3

2.2. Sample Preparation

The sodium hydroxide solution and the aqueous sodium silicate were stirred for 15 min by a magnetic stirrer. After that, the AAB specimens were manufactured by adding the activator solutions to the solid precursor (coal fly ash. granulated slag and aggregates). Then, all specimens were produced by mechanical mixing for 4 min. Subsequently, the AR-glass fibres were added to the fresh AAB and mixed together for 0.5 min. The detailed mixing proportion properties are shown in **Table 3**.

For the structural study of hardened AAB, the specimens were cast into corresponding molds and cured at a temperature of 20°C and a relative humidity of 65% for 24 hours. Following this, the samples for the tests to be performed on the hardened concrete were stored dry, according to EN 12390-2.

2.3. Test Set-Up

The 3-point bending tensile strength was determined by means of the Toni Technik ToniNorm (load frame 20 kN) with three samples measuring $160 \times 40 \times 40 \text{ mm}^3$ (length \times width \times height), based on EN12390-5 (**Figure 1(a)**). The compressive strength was determined by means of the Toni Technik ToniNorm (load frame 3000 kN) following EN 12390-3, with three cubes having an edge length of 150 mm (**Figure 1(b)**). The pre-load and the loading speed were 18 kN and 0.5 mm/min respectively.





Figure 1. Determination of 3-point bending tensile (a) and compressive strength (b).

Table 3. Composition of AAB and OPCC in wt%.

	Component	Mixing technology	Mixing power in %	Mixing time in s
1st	Binders + aggregates	concurrent	15	60
2nd	Activator solution + solid precursor	sequence	40	240
3rd	AR-glass fibres	sequence	40	30

To quantify the durability of the AAB-matrix, the freeze-thaw test, the water permeability and the acid resistance were investigated. The freeze-thaw test was measured with the CIF-Test (Capillary suction, Internal damage and Freeze-thaw test) by the Schleibinger Freeze-Thaw-Tester with standard agent solution according to the recommendations of RILEM TC 176 IDC (Figure 2(a)). The acid resistance was determined by the mass loss after storage of three specimens (28 days old) in 37% hydrochloric acid for 7 days at a temperature of 21°C (Figure 2(b)). The determination of the water permeability was carried out according to DIN EN 1062-3 (Figure 2(c)).

3. Results and Discussion

3.1. Properties of AAB

Table 4 reveals the fresh and hardened AAB characteristics at the age of 14 days. Using an air content tester, a gross geometric density of 2.39 g/cm³ and air content of 2.4 volume percent and were determined in the fresh AAB. The 3-point bending tensile and compressive strengths of the hardened AAB were 5.5 and 53.4 MPa respectively. The pH-value of the fresh AAB was 14, whereby the pH-value decreased to 10 due to the reactions in the hardened AAB. A dynamic elastic modulus of 36 GPa was determined by the ultrasonic examination. The total shrinkage deformation was 0.45 mm/m.

Furthermore to the reactivity and the solids content of the alkali activated binders, the amount and concentration of the alkaline activator have an influence on the solubility and reactivity of the AAB [38]. For this influence, the 3-point bending tensile

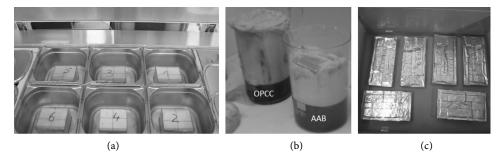


Figure 2. (a) CIF-Test; (b) Acid storage; (c) Water absorption.

Table 4. Basic properties of fresh and hardened AAB (NaOH: 5.5 mol/l).

	Fresh AAB	Hardened AAB	
Geometric bulk density	2.39 g/cm ³	2.35 g/cm ³	
pH-value	14	10	
Compressive strength	-	53.4 MPa	
3-p. bending tensile strength	-	5.5 MPa	
Elastic modulus (dynamic)	-	36 GPa	
Total shrinkage	0	0.45 mm/m	

strength (after seven days) is shown as a function of the NaOH concentration (1.2 to 10.0 mol/l) in **Figure 3**. Referring to **Figure 3**, an increase of the sodium hydroxide concentration leads to an increase of the 3-point bending tensile strength. The sodium hydroxide concentration of over 5.5 mol/l did not further increase the 3-point bending tensile strength, because the saturation point with respect to the solubility of the binder was reached, thus leading to a high level of degree of reaction. Sodium hydroxide concentrations over 5.5 mol/l did not result in an increasing of the 3-point bending tensile strength after seven days.

3.2. Thermogravimetry

The thermogravimetry and the differential thermogravimetry of the AAB-matrix are illustrated in Figure 4. Regarding to Figure 4, it can be observed that the weight loss started at a temperature of 35°C with maximum weight loss at 87°C. This weight loss can be attributed to the removal of free and unbound absorbed water molecules. The further mass loss up to a temperature of about 300°C corresponded to the physically bound water in the porous matrix and the zeolite-water from the nanocrystalline zeolite structures [39] [40]. The mass loss up to a temperature of around 520°C resulted from the decomposition of the chemically bound water from the calcium silicate and the calcium aluminate phases. The cumulated weight loss was of 10.5% at a temperature of 900°C.

3.3. Durability of AAB Matrix

The results of the durability of the alkali activated binder were investigated and the comparison with the ordinary Portland cement concrete is shown in **Table 5**. The results comprise the freeze-thaw test, the water permeability and the acid resistance. The AAB-matrix had a water absorption coefficient (w_t) of 0.095 kg/m²·h^{0.5} and reached the class 3 (low water absorption) according to DIN EN 1062-3 (**Table 5**). As opposed to this, the OPCC had a water absorption coefficient of 1.301 kg/m²·h^{0.5} and reached the class 1 (high water absorption).

Table 5. Results of the durability of AAB and OPCC.

	AAB	OPCC
Water absorption coefficient		
$w_t \text{ in kg/m}^2 \cdot h^{0.5}$	0.095	1.301
Class (DIN EN 1062-3)	3	1
Acid resistance		
Weight loss in %	15.9	75.1
CIF-Test		
Ru, ₂₈ in %	96	91
m_{28} in g/m^2	30	217

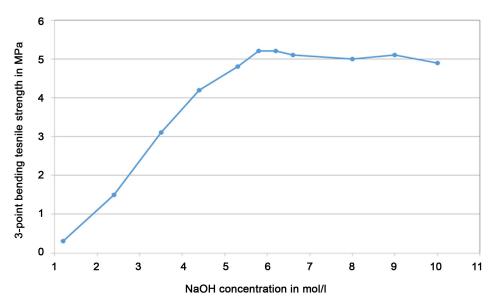


Figure 3. 3-point bending tensile strength as a function of the NaOH concentration.

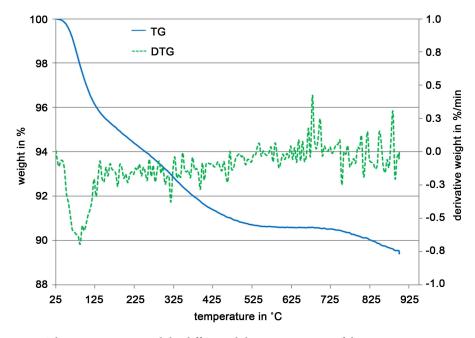


Figure 4. Thermogravimetry and the differential thermogravimetry of the AAB.

In addition, the AAB-matrix exhibited a high frost resistance, which was verified through a successful CIF test with a relative dynamic modulus of elasticity of 93 percent (Ru, 28) and an average scaling of 30 g/m 2 (m $_{28}$) after 28 freeze-thaw cycles (**Table 5** and **Figure 5**).

After seven days storage in 37% hydrochloric acid, the AAB-matrix had a mass loss of 15.9% (see **Table 5** and **Figure 6**). In comparison, the mass loss of the OPCC was significantly higher with 75.9%. The high acid resistance of the AAB-matrix is mainly due to the portlandite-free phases.

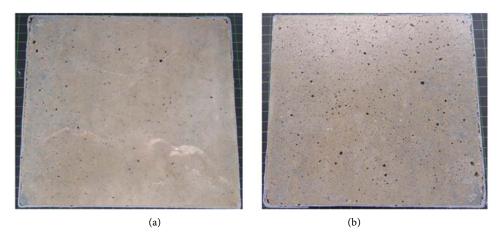


Figure 5. Test surface before (a) and after (b) the CIF-Test.



Figure 6. AAB and OPCC samples before (left) and after the acid attack (right).

4. Conclusions

In this study herein, the development of an AAB-matrix and the performance and durability of different fibres in the AAB-matrix were investigated through various experimental work. Based on the results of this study, the following key points are drawn:

- The adapted molarity of the sodium hydroxide solution was 5.5 mol/l. Sodium hydroxide concentrations over 5.5 mol/l did not result in an increasing of the 3-point bending tensile strength after seven days,
- The AAB achieved a compressive and a 3-point bending tensile strength of 53.4 and 5.5 MPa respectively after one day at ambient temperatures,
- The acid resistance of the AAB was 500% higher than the ordinary Portland cement concrete, which could be attributed mainly to the free portlandite phases.

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