Properties control in autoclave aerated concrete by choosing of pore forming Al-agent

E V Fomina¹, A S Chulenyov² and N I Kozhukhova³

¹PhD in Engineering sciences, Belgorod State Technological University named after V.G. Shoukhov, 308012, Belgorod, Kostukov Street, 46, Russia.
²PhD in Engineering sciences, National Research University Moscow State University of Civil Engineering, 129337, Moscow, Yaroslavskoye Shosse, 26, Russia.
³PhD in Engineering sciences, Belgorod State Technological University named after V.G. Shoukhov, 308012, Belgorod, Kostukov Street, 46, Russia.

Abstract. Autoclave aerated concrete is state of art high-performance construction material due to unique combination of thermal and strength characteristics achieved by cellular structure, in general. In this paper effect of dispersity and reactivity of gas forming Al-component on carrying out of such important processes as forming of optimal pore structure providing reduced density and required compressive strength in aerated concrete. The results of kinetics of gas evolution in different alkali media in the binder under temperature and effect of Al-component were obtained. Volume of gas evolution was measured with gasometric method. This method is based on variation of volume of hydrogen evolution caused by reaction between Al-component and Ca(OH)₂. According to experimental and calculated data it is reasonable to use polydispersed Al-component containing nanoparticles. It allows realization gradual gas evolution, directed formation of required shape and size of pores in cellular structure leading to reduction in density by up 16% and reducing in water absorption capacity by 41% as well as increasing of compressive strength by 28% and increasing of humidity resistance.

1. Introduction

In conditions of state of the art construction the highest influence has solution of two interacting problems: enhancement of thermal characteristics of envelopes and reducing of material consumption. One of the effective ways to solve this problem can be application of aerated concrete. Light-weight cellular concrete has the following advantages in buildings: high heat- and sound-insulation, good workability, dramatical reducing of load on load-bearing elements, saving the required strength and reducing a labor cost etc. [1]. Among wide range of cellular concrete the most popular is autoclave aerated concrete (AAC) block [2, 3].

Cellular structure of aerated concrete provides a favorable microclimate for human life and activities. The main problem of high-performance aerated concrete production with high porosity and high compressive strength is providing of favorable conditions for joint caring out of two chemical processes: gas emission and gas keeping. It is necessary to superpose a rate of gas emission and rate of increasing of structural viscosity of aerated mixture [4, 5]. At the same time a gas emission process should be finished before initial setting of the binder [6, 8]. Simultaneous realization of these requirements can be achieved by correct choice of gas forming agent. In earlier studies [8, 9] was reported that granulometry of Al-component is important to achieve the optimal thermal and physical-mechanical characteristics. The most preferable particle size is smaller than 50 µm, providing a finely porous structure and forming a lower density at the same strength in cellular composite.
Normally, the most usable gas forming agent for AAC block is Al-powder. But it has the following disadvantages: low reactivity, high dusting level, necessity of surfactant application, risk of fire and explosion. Therefore, it is better to use Al-paste to avoid the above characteristics. State of art high-performance Al-paste meeting requirements in granulometry are expensive and has a high effective concentration in concrete [10]. Fast growing market of high-performance AAC block initiates a force requirement to gas forming agents. In this case the submicro- and nano-sized particles are desirable.

Studies on application of nanodispersed gas forming agent (NDG) in aerated concrete were accomplished earlier [11]. NDG is activated aluminum with particle of 80 nm enclosed by polyethyleneglycol. High-dispersed Al-particles form low-strength conglomerates which decompose in aqua medium. So, dusting not takes place. NDG, generally, is used for hydrogen production. To extend the application fields the heat-insulating aerated concrete with enhanced characteristics was developed [12]. Composition and production technology for AAC block has significant differences. For example, AAC block has a lime component effecting on pH-value and viscosity of binding mixture [13]. Object of this study is the formation of optimal porosity, decreasing in density but saving the same strength values.

2. Experimental section

Dispersity of gas forming agent was estimated according to Blaine fineness determined by Blain machine PSH-12; granulometry was measured by laser diffraction method with MicroSizer 201C machine. Microstructure was studied with scanning electron microscope TESCAN MIRA 3 LMU.

Taking into account parameters of production of the AAC block with enhanced characteristics the apparatus for gasometric method was prepared for detail investigation of gas emission process (Figure 1). The experimental aerated mixture with required temperature was placed into heat-resistant glass with magnetic stir bar which allows simultaneous stirring and keeping the working temperature a constant. The glass was hermetically capped by cap with pipe for a gas disposal. Flask with three necks was a reservoir for water, replaced by the forming gas.

![Figure 1. Scheme of apparatus for gasometric method](image)

1 – heat-resistant glass; 2 – magnetic stirrer; 3 – reservoir with water; 4 – measuring cylinder

In this work two types of gas forming agents: Al-paste GPB-1 (Logosib Company, Russia) and nanodispersed gas forming agent (NDG) (Ecoenergotech Company, Russia) were used. Weight percentage of aluminium in these was more than 80%.

3. Results section

3.1. Particle size analysis

According to data of Blain method the specific surface area (SSA) for GPB-1 was 693 m²/kg, that was lower vs. NDG – 843 m²/kg.

Grain-size analysis demonstrated that GPB-1 was monodisperse in size range of 0.35–33 µm with max particle concentration in area of 3–4 µm (Figure 2).
NDG was polydisperse component with particle in the following size ranges: 0.9–2; 2.75–3.63; 6–7; 11–13 µm. Also, NDG contained particles lower than 0.2 µm, including nano-sized particles.

3.2. Microstructure
Sceleton frame of GPB-1 consists of flakes with width of 30 µm, and thickness of 3–4 µm.

Polydisperse composition of NDG is characterized by average particle size of 3 µm and particle thickness of 90 nm. It seems presence of nano-sized particles, characterized by self-assembly leads to formation of high-density packing.

3.3. Gasometric method
The main indicator of reactivity of gas forming Al-component is basicity of reaction medium in the binder where gas evolution is realized. As a binder, a cement and unslaked lime were used. Therefore the kinetics of gas emission was studied using saturated solution of calcium hydroxide [14] obtained from cement and lime components. Two types of gas forming agents separately were added to water to obtain a suspensions followed by introduction of these to saturated alkali solution. The saturation of
solutions was realized when the lime slaking and the cement hydration due to Ca²⁺ ions emission. So, lime component formed a solution with pH of 12.50 and a cement component – 10.70. Other main parameter was temperature. It was a constant (T = 40°C) according to the production conditions. During the gas emission procedure the stirring of solutions took place.

**Table 1.** Kinetics of gas evolution in different conditions

| Component of alkali solution | Gas forming agent | Temperature, °C | pH  | Volume of gas emission, m³ | Rate of gas emission, sec |
|-----------------------------|-------------------|-----------------|-----|---------------------------|---------------------------|
| Lime                        | NDG               | 25              | 12.5| 8×10⁻⁶                    | 360                       |
|                             |                   | 40              |     | 55×10⁻⁶                   | 660                       |
|                             | GPB-1             | 25              |     | 15×10⁻⁶                   | 140; 390                 |
|                             |                   | 40              |     | 50×10⁻⁶                   | 860                       |
| Cement                      | NDG               | 25              | 11.7| 5×10⁻⁶                    | 60                        |
|                             |                   | 40              |     | 35×10⁻⁶                   | 700                       |
|                             | GPB-1             | 25              |     | 5×10⁻⁶                    | 100                       |
|                             |                   | 40              |     | 9×10⁻⁶                    | 230                       |

Data from Table 1 demonstrates the maximal gas emission for two types of gas forming agents took place for pH = 12.5 (in lime based solution) and at 40°C. According to [8] the maximal rate of gas emission should be realized when a lime slaking period. As in this work a lime with reactivity of 85% and slaking period of 8–12 min, the most effective gas forming agent was NDG with maximal volume of gas emission of 55×10⁻⁶ m³ realized at 11th minute of slaking.

For more deep study the volume of gas emission was measured every 10 sec (Figure 4).

**Figure 4.** Kinetics of gas emission of:
1 – NDG; 2 – GPB-1; 3 – NDG at T = 40°C; 4 – GPB-1 at T = 40°C

Stirring procedure is important when interaction of AI-component and the binding mixture with basic pH-value.

Normally, reactive Al-particles are enclosed by organic component. When interaction of these particles the organic component was destructed and dissolved, increasing reactivity of Al-particles and increasing of volume of gas emission (Figure 4, the peak at 1th minute for two types of gas forming agents).

For GPB-1 a short-term intensive gas emission is typical. That is negative effect leading to destruction of structure of hardened concrete such as pore defects and structural cracks. Also, two peaks were observed. One of these was fixed at 1–2 minutes of gas emission leading to negative loss of the formed gas. At ambient temperature the gas emission for GPB-1 and NDG was finished at 10th and 8th minute, respectively. Increasing of temperature increases the reactivity but does the gas...
emission process longer. In this case the gas emission for GPB-1 and NDG was finished at 16\textsuperscript{th} and 21\textsuperscript{th} minute, respectively.

Polydisperse composition of NDG allows gradual gas emission. Maximal volume of gas emission ($55 \cdot 10^{-6} \text{ m}^3$) was realized at 11\textsuperscript{th} minute and at 40°C followed by decreasing of gas emission rate by up $1 \cdot 10^{-6} – 2 \cdot 10^{-5} \text{ m}^3$ up to end of the process.

3.4. Characteristics of aerated concrete after autoclave treatment
As a reference composition was an aerated concrete with density of 500 kg/m\(^3\) and compressive strength of by up 2.5 MPa. Basic raw materials were meet requirements to Russian Standard 31359-2007. Ratio mixture provided density in concrete with density of 500 kg/m\(^3\). Experimental samples were cubes of 100×100×100 mm. Autoclave treatment of the fresh specimens was performed under the following curing regime: air-flush of the specimens in the curing chamber for 40 min; after that pressure was buildup by 10 atmospheres and temperature up to 183°C during 1 hour. The curing process was performed at constant 10 atmospheres and 183°C for 5 hours followed by pressure release during 2 hours.

**Table 2.** Physical and mechanical characteristics of AAC

| Composition | Compressive strength, MPa | Density, kg/m\(^3\) | Water absorption, % | Gas keeping capacity $K_k$ at $t = 40°C$ | Coefficient of pore deformation $K_{def}$ at $t = 40°C$ |
|-------------|--------------------------|---------------------|---------------------|------------------------------------------|-----------------------------------------------|
| 1           | 2.9                      | 530.65              | 70.60               | 0.07                                     | 1.89                                          |
| 2           | 3.7                      | 445.84              | 41.75               | 0.12                                     | 1.68                                          |

According to data from Table 2, density of the reference composition was 530.65 kg/m\(^3\) and compressive strength was 2.9 MPa. Using of NDG allowed reducing of density in AAC up to 445.84 kg/m\(^3\) and increasing in compressive strength up to 3.7 MPa.

![Figure 5](image1.png)  
*a* – composition 1; *b* – composition 2 (Table 2)
Macrostructure of reference AAC based on GPB-1 was characterized by irregular pore shape, a deformed interpore partition, difference in pore size as well as presence of interconnecting pores (Figure 5 a). Coefficient of pore deformation $K_{def}$ was 1.89 (Table 2, Equation 3). In this case the structure of AAC absorbed water more quickly and keep it inside with water absorption of 70.60% (Table 2, Equation 1). The highest pore content was with size of 160 μm – 1 mm.

Polydisperse NDG with average particle size of 3 μm reduced a pore size in 2–2.5 times providing a pore size of 50–500 μm (Figure 5 b). In this case AAC block was characterized by finely-porous structure with homogeneous distributed spherical closed pores and with reduced $K_{def}$ up to 1.68 (Table 2). Closed porosity provided reducing of water absorption by up 41%.

3.5. Calculated porosity of autoclave AAC block

Water absorption of AAC with an accuracy of 0.1% was calculated by the following:

$$ W = \frac{m_{water} - m_{dry}}{m_{dry}} \times 100\% , \text{ where} $$

$m_{water}$ – weight of water-saturated sample, g;

$m_{dry}$ – weight of dried sample, g.

When calculation of volume of gas emission it should be taken account that 1 kg of Al-paste produces 1.245 m³ of gas under standard conditions according to the following reaction:

$$ 3\text{Ca(OH)}_2 + 2\text{Al} + 6\text{H}_2\text{O}_3 \rightarrow \text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{H}_2\text{O} + 3\text{H}_2\uparrow $$

Increasing of temperature lead to increasing of volume of gas emission. For example, at 40°C gas emission achieved to 1.425 m³. Under real production conditions the Al-paste consumption is greater due to the present of part of unreacted aluminum as well as due to loss of some of the aluminum when stirring of the aerated concrete. It can be investigated by gas keeping capacity $K_k$. $K_k$ coefficient was calculated as ratio between volume of kept gas ($V_k$) and volume of gas emission ($V_e$).

Gas keeping capacity $K_k$ was calculated by following:

$$ K_k = \frac{V_k}{V_e} $$

Coefficient of pore deformation $K_{def}$ for AAC was determined by following:

$$ K_{def} = \frac{1}{n} \sum \frac{d_1}{d_2} , \text{ where} $$

$d_1$ – the biggest pore diameter, mm;

$d_2$ – the smallest pore diameter, mm;

$n$ – number of pore, units.

Conclusions

The main parameters effecting on rate of gas emission are dispersity and SSA of Al-particles as well as pH-value and temperature of reaction medium. Application of NDG containing the particle smaller than 0.2 μm as well as nanoparticles allows carrying out gradual gas emission and keeping of gas into the concrete massive. Increasing in volume of gas emission leads to increasing of porosity and reducing in density by 16%. Presence of differently-sized closed pores in range of 50–500 μm, homogeneously distributed in the aerated composite provides increasing in water resistance of AAC block by 41%. Formation of free-of-defect structure provides increasing in compressive strength by 28%.

So, variation of pore structure of AAC block by application of polydisperse gas forming agent the formation of required structure in compliance with operation conditions is possible. Under production
conditions it allows the variation of technological cycle duration, parameters of final product as well as reducing of product failure.

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