Units for accelerated carbonation of concrete samples and repair compositions

V M Latypov, P A Fedorov, E V Lutsyk and T V Latypova

Ufa State Petroleum Technological University, 1, Cosmonauts Str., Ufa, 450062, Russia

E-mail: stexpert@mail.ru; fpa_idpo@mail.ru

Abstract. The article is devoted to the modernization issues of laboratory units designed for the concrete carbonation study. General pre-conditions for accelerated tests as well as developments of respective units are given. The article provides a rationale for the choice of carbon dioxide volume concentration and temperature and humidity conditions in a chamber. The article describes some variants of units with indication of their pros and cons and introduces a unit for carbonation of concrete with a humidifier and automatic control system.

1. Introduction

Enhanced concentration of acid gases in the Earth’s atmosphere and especially of carbon dioxide (henceforth CO₂) leads to the decrease in the service life of reinforced concrete structures because of excessive wear. One of the main reasons for it is carbonation of concrete. Under this process it is customary to understand a complex physicochemical process of neutralization of hydrated phases of cement matrix by means of CO₂ as well as alteration of alkalinity of the pore liquid from pH 12 to pH 8.5 [1]. Alkalinity reduction is not a harmful factor for concrete. However, it becomes critical for reinforcement corrosion when in the area of its location we can observe chlorides, access of oxygen, water penetration, potential difference and other factors to which reinforced concrete structures are exposed. [2-4]. As a result, protective properties of concrete are lost, and steel reinforcement corrosion begins. When increasing in volume, reinforcement corrosion products destroy the structure of the protective concrete layer which leads to the loss of bearing capacity of the construction. Carbonation of concrete proceeds more intensively in constructions which have cracks with width of more than 0.1…0.2 mm, as well as other defects and damages that reduce the depth of concrete cover.

At present time there is no single approach to the prediction of durability of reinforced concrete structures from the perspective of CO₂ influence. This is connected with the complexity of capillary-porous structure of different types of concrete, variability of temperature and humidity operation conditions and influence of a large number of other factors. It is obvious that to study the process of concrete carbonation as well as to obtain kinetic dependencies it is necessary to develop optimal research methods as well as special units allowing to simulate the influence of operational environment without distorting the results.

2. General pre-conditions for development of laboratory research methods

It is a common practice to study carbonation of concrete in two directions – full-scale tests and laboratory tests. Full-scale tests are of two types: testing of control samples in operational environment...
and inspection of construction being in service during a certain period of time. Installation of control samples in operational environment requires long-time measures to achieve the necessary amount of values that describe the corresponding kinetics. For example, according to publication [5], approximately 9-10 months are required to achieve carbonation depth of several millimetres for concretes with low permeability and by gas concentration not more than 0,04% by volume. When it comes to examination of structural unit during its service life, the formed database of concrete carbonation values includes one, more rarely two figures. This necessitates to consider a wide range of kinetic dependencies. Besides, these values are drastically influenced by variable temperature and humidity operation conditions of the construction as well as uneven distribution of CO$_2$ concentration. For instance, if the construction was periodically humidified during its operation, the slowdown of carbonation happened because of pore blockage with moisture. Thus, in publication [6] the data are given that carbonation indoors is higher than outdoors. Measurement results of CO$_2$ concentration inside the building given in publication [7] show that in the presence of wind penetrating through interstices, concentration values indoors vary within the range of 20-30%.

Nowadays the majority of researchers give priority to laboratory tests. Their main advantage is high speed of creation of the required database of values for carbonation depth of the test samples, which allows to simulate the kinetics of the given physicochemical process. It happens owing to densification of CO$_2$ that leads to acceleration of concrete carbonation. Upon that, increase in gas concentration should not violate the diffusive mechanism of carbonation inhibition because of possible appearance of Stefan flow. At present the issue about a limiting value of this concentration remains open to question. Thus, in publication [5] it is pointed out that at concentration below 30% by volume the influence of Stefan flow can be neglected.

In Russia limit value for CO$_2$ concentration is standardized by government standard GOST 31383-2008 “Protection of concrete and reinforced concrete structures from corrosion. Test methods” and is equal to 10%. In EU countries this value is regulated by BS EN 1329:2004 “Products and systems for the protection and repair of concrete structures. Test methods. Determination of resistance to carbonation” and is equal to 1% by volume. However, at 10% concentration of CO$_2$ carbonation process may proceed with excess gas amount, and in this case, it is controlled only by diffusion. But in natural conditions at 0,04…0,05 % concentration of CO$_2$ this process proceeds with gas shortage, that is why it is already controlled by two parameters: diffusion and chemical reaction (figure1).

![Figure 1](image_url)

**Figure 1.** Diagram of concrete carbonation in conditions of its exposure to different concentrations of carbon dioxide: a - in natural environment with 0,05% concentration of carbon dioxide; b – during accelerated tests with 10% concentration of carbon dioxide.

Moreover, according to publication [9], at 1% concentration of CO$_2$ by volume the same
carbonation products are formed as in natural conditions. Taking these data into account the value of 5% concentration of CO$_2$ by volume is taken as a working one in the unit for accelerated testing.

The choice of optimal relative humidity of gas-air environment for standard tests is also debatable. Generally, its value is in the range from 55 to 80%. It should be noted that in this range the highest carbonation rate is observed, and inhibition is missing. In document BS EN 13295:2004 value 60% is accepted as a standard value for humidity. On the other hand, according to GOST 31383-2008 standard relative humidity should be equal to 75%. Choice of this value is connected with the fact that at about 80 % humidity intensive steel corrosion is observed [5]. It is necessary to indicate that humidity value of 60% corresponds to the normal operation and 75% - to wet operative conditions. Therefore, in the second case the process proceeds with inhibition and it will have an effect on kinetic dependencies.

To simulate temperature and humidity conditions in the unit under development value 75% for humidity is accepted.

3. Constructions of units for accelerated carbonation of concrete

One of the earliest proposed unit modifications for the study of concrete carbonation was a test bench that included desiccator, burettes, Tishchenko flask and other elements. A sample was placed into a desiccator and gas-air mixture with CO$_2$ was supplied [10]. So far as CO$_2$ was absorbed by the sample, its concentration decreased and in the process of the test a new portion of gas was added manually. Concentrations 100; 12,5 and 12 % by volume were taken as working ones. Humidity of the mixture was regulated by means of distilled water or sulfuric acid of different concentration. Burettes with water were additionally mixed with a small amount of paraffin oil to prevent water fixation with CO$_2$.

Imperfection of the first unit modifications, small amount of test samples as well as the growing interest of research community in the study of concrete resistance in conditions of CO$_2$ influence contributed to the further development of this research area. And thus, a wide range of upgraded units appeared which are described in publications [5, 11, 12, 13, 14, 15]. Not without interest is a unit introduced in Japan and made in the form of wood-frame gas chamber in which test samples were placed on racks [14].

There exists a unit for carbonation of concrete samples given in publication [11]. This unit consists of three parts: balloons with CO$_2$ and clean air; a system for gas-air mixture producing with 50% CO$_2$ concentration by volume and with required humidity; a gas chamber with installed samples and a system of supply and removal of exhausted gas-air mixture. Gas-air mixture of the required concentration and humidity was obtained by means of the installed saturator and desiccator. Gas-air mixture was controlled with the help of flow rate meters. The main advantage of this unit is that gas-air mixture is obtained with more accurate parameters since there are no admixtures and aerosols and that it is possible to test a large number of samples of different types. The main disadvantage of this unit is pointwise supply of gas-air medium to the samples as a result of which it is necessary to regulate the flow of the mixture as well as nozzle location in relation to the sample.

Publication [16] describes two units with manual and automatic control. Their distinctive feature is as follows: to achieve humidity of 77% it was proposed to use a saturated solution of NaCl which was placed in the desiccator with the sample. In addition to it there was a manometer in this unit, which was needed for monitoring of air rarefaction while CO$_2$ absorption in the chamber. 10% concentration of CO$_2$ by volume was accepted as a working one. For automated control a two-sensor system was installed in a metering vessel. When the channel of the metering vessel is filled with water the lower sensor closes with water. As a result, a gas valve closes. A significant drawback of these units was that most commonly only one sample can be used. To test a group of samples, units with both manual and automated control were presented in publication [5]. The significant difference of these units was availability of a fan for gas and air mixing. The mixture was created directly in the chamber where the samples are located.
Publication [17] describes a manually operated stand, the prototype of which was an upgraded version of the unit described in publication [5]. The stand consists of the following main parts: sealed chamber (V=0.2 m³); carbon dioxide bulb; gearbox; automatic gas analyzer “OKA-T-CO₂”. The required humidity in the unit was obtained by means of the tub with a saturated solution of NaCl. CO₂ was supplied to the chamber through the pipe installed in the cover. A flexible sleeve with incurved end and grid cutting the flow with 1 mm cells was installed in the end of the pipe to prevent a single-point impact of CO₂ flows on the samples. The measurements of CO₂ concentration height wise the chamber showed that at the gas supply level the concentration reached approximately 10…15% and in the area with NaCl solution tub it was only 1…3%. The average concentration in the zone of sample installation was equal to 5% by volume. To prevent uneven carbonation of concrete the unit was supplemented with the system of low-speed fans which were installed inside the tank from below along the contour. Excessively high fan speed can create artificial gas injection to the pores of the samples which will lead to the distortion of the results. The optimal propeller speed should be equal to 800 rpm – which corresponds to the class of low-speed fans. Excessive pressure in the chamber is relieved through the installed in the side wall U-shaped tube filled with water.

There are two essential drawbacks in the described unit: a limited number of test samples and lack of automated control system. Bearing this in mind we developed a modified unit (figure 2). It allows to carry out accelerated tests of a large number of samples by various CO₂ concentration. Gas supply in this unit is carried out by means of a gearbox with distribution manifold. There is a separate sleeve with grid that cuts the flow of the supplied mixture inside each chamber. To control CO₂ concentration a hole with a sealed cover was made in each chamber. Gas analyzer «OKA-T-CO₂» is lowered through this hole. Humidity in each chamber was measured with the help of autonomous temperature and humidity logger “EClerk-M-RHT” with memory system for recording and data transfer to a computer.

Since 2010 the authors have been searching for the optimal parameters of the unit which will allow to increase accuracy of test results as well as to carry out simultaneous testing of a large number of samples. They proposed two concepts of the unit with wired and wireless control system allowing to control carbonation of concrete at a distance on a real time basis.

In 2016, based on these concepts, the authors of this research developed a unit shown in figure 3.
The difference of this unit is that in addition to the availability of automated control system of gas supply to the chamber there exists an opportunity to promptly change temperature and humidity conditions and continually record all the variables of the environment inside the chamber. Gas supply to the chamber is carried out through the flexible sleeve with cutting grid. To maintain humidity inside the chamber we installed an ultrasonic humidifier instead of a sodium chloride tub – to increase relative humidity and dehumidifier – to reduce relative humidity. All the processes are controlled by programmable logic controller “PLK 73” which receives data from gas analyzer sensor “PKU-4/1-Shh”. Upon reaching concentration threshold values the controller transfers information to the electromagnetic valve mounted on the CO$_2$ bulb.

![Figure 3. Unit for accelerated tests with automatic feeding: 1 – automated control system block including controller and gas analyzer; 2 – sealed «reaction» tank (V=0,2 m$^3$); 3 – system of low-speed fans; 4 – electromagnetic valve; 5 – samples; 6 – rack for samples placement; 7 – ultrasonic humidifier of gas-air environment; 7.1 – fan; 7.2 – exhaust outlet; 7.3 – radiator system; 8 – CO$_2$ concentration analysis sensor “PKU-4/1-Shh”; 9 – gas-air environment temperature and humidity sensor “EClerk-M-RHT”](image)

It should be pointed out that similar units in different modifications exist in China. As a rule, they are implemented on the basis of climatic chambers of medium and large volume. They are additionally equipped with gas supply system and gas analyzers. However, by large volumes of chambers there is a possibility of uneven carbonation which contorts kinetic dependencies. That is why it is necessary to pay attention to the reasonable number of fans as well as to gas supply system.

4. Conclusion
On the basis of the conducted research in test methods of carbonation of concrete and repair compositions we have developed a unit allowing to perform tests at different CO$_2$ concentration with a large number of samples. The unit has a special gas supply system that excludes its single-point impact on the sample. It is achieved due to the installation of flexible sleeve with the grid cutting the flow. More than that, it allows to mix gas-air environment more evenly because of mounting of low-speed fans with propeller speed 800 rpm in lower levels.

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