



Characterizing gas bubble size distribution of laboratory foamed cement using X-ray micro-CT

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HIGHLIGHTS

- A micro-CT investigation of foamed cement produced in sealed mixers was conducted.
- Gas bubble size of foamed cement studied generally follows the normal distribution.
- The mean of the distribution increase exponentially with foam quality.
- The standard deviation of the distribution increase exponentially with foam quality.
- Applied pressure significantly reduces gas bubble size in foamed cement.

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ABSTRACT

The objectives of this study are to use micro-computed tomography (micro-CT) to elucidate the influencing factors of the microstructure of laboratory foamed cement and develop statistical models to quantify the gas bubble size distributions. During this study, foamed cement slurries were prepared using sealed foam cement mixers at various operating conditions. The influences of shear rate, mixing energy, base cement slurry composition, blender geometry, and applied pressure on the gas bubble size distribution of set foamed cement were investigated. Test results indicate foam quality and gas pressure are the primary determining factors of gas bubble size in foamed cement. The gas bubble size in foamed cement produced by a sealed foam mixer approximately follows the normal distribution. For samples generated with a standard multiblade blender at atmospheric condition, the mean and standard deviation of gas bubble size distribution increase exponentially with increasing foam quality. A dramatic decrease in gas bubble size is observed with increasing gas pressure. On the other hand, variations in mixing energy, shear rate, base slurry composition, and blender geometry have relatively little effect on the microstructure of the foamed cement within the range investigated.

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1. Introduction

Foamed cement is created by stabilizing gas bubbles in a cement matrix by the use of a foaming agent. Since its invention in the 1980s [15,6], foam-cementing has found many applications in the oil and gas industry despite of the higher operational cost than conventional cement systems. Foamed cement has many advantages compared to the conventional cement systems. Some of the most prominent benefits associated with foamed cement are its abilities to improve mud displacement [5], mitigate loss circulation [9,4,3], and improve long-term well integrity [4,10,16]. The mechanical performance and other properties of foamed cement are directly associated with its microstructure, which is

dependent on the method of foam generation [8,13]. However, the exact correlations between the microstructure foamed cement and the various parameters during foam generation such as shear rate, mixing energy, base slurry property, and applied pressure are poorly understood. The lack of proper experimental technique to fully characterize the properties of foamed cement has somewhat limited its application.

In recent years, X-ray micro-computed tomography (micro-CT) technology has been used to analyze the porosity of building materials such as concrete at different resolutions [7]. X-ray micro-CT has also been proven to be an effective method to evaluate the microstructure of foamed cement and provide reliable quantitative characterization of the material in terms of gas bubble size distribution [11,12,14]. X-ray micro-CT can provide excellent visualization and detailed quantitative information about the gas bubbles in foamed cement, which cannot be obtained by simple density

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measurements. Recent studies have shown that that foamed cement produced by a multiblade Waring® blender differ significantly from that produced by field equipment in gas bubble size distribution due to the fundamentally different methods of foam generation [14]. Nevertheless, it is necessary to obtain a more complete understanding of foamed cement produced by Waring® blender because it is the not only the most widely used method of generating foamed cement in a laboratory environment but also the current industry standard for laboratory evaluation of foamed cement (API 10-B).

A comprehensive experimental program is initiated here to investigate the relationships between the operational variables of laboratory foaming process and the microstructure of foamed cement, particularly gas bubble size distributions. A standard multiblade Waring® blender and a multiple analysis cement system (MACS) were both employed to generate foamed cement. The main focus of this study was on the influences of foam quality, shear rate, and mixing energy on the gas bubble size distribution in foamed cement. Other influencing factors of the gas bubble size distribution in foamed cement, such as base slurry property, applied pressure, and blender geometry were also preliminarily investigated. Statistical models were fitted to experimental data to derive mathematical representations of gas bubble size distribution in foamed cement. As discussed by [14], the gas bubble size in foamed cement produced by Waring® blender generally follows the normal distribution described by Eq. (1):

$$f(D) = \frac{1}{\sqrt{2\sigma^2\pi}} \exp\left(-\frac{(D-\mu)^2}{2\sigma^2}\right) \quad (1)$$

where D is the gas bubble diameter; $f(D)$ is the volumetric probability density function for gas bubbles of diameter D ; μ and σ are the mean and standard deviation of D , respectively.

2. Experimental method

The base slurry used in this study was neat Class A cement mixed with 45.3% water by weight of cement (bowc). The liquid density of the base slurry was calculated to be 15.7 lbm/gal. In preparation of the foamed cement slurry, the base slurry was first prepared following API standard procedures (API RP 10B-2 [1]). Then, appropriate amounts of base slurry and surfactant (foaming agent) were added to a foam blender to produce foamed cement. The dosage of surfactant was applied at 2% by volume of water (bvow) in the base slurry. It should be noted that the dosage of surfactant has no impact on the microstructure or mechanical property test results of foamed cement when varied in the range from 0.5% to 6% bvow [13]. American Petroleum Institute (API) specifies that the slurry should be mixed in a sealed foam blender at the 12 000 rpm (r/min) setting for 15 s (API RP 10B-4 [2]). During this study, in order to look into the effect of shear rate and mixing energy on the properties of the foam cement, both rotational velocity and mixing time were varied, and the combinations used are shown in Table 1. The foam qualities investigated include 20%, 30%, 40%, 60% and 80%. It should be noted that when the 12000 r/min setting was used, maximum attainable r/min was approximately 6000 at 20% foam quality and 7000 at 60% foam quality.

Table 1
Combinations of mixing parameters used.

r/min setting	1000	1000	1000	4000	4000	12000	12000
Actual r/min	1000	1000	1000	4000	4000	6000–7000*	6000–7000*
mixing time (s)	15	120	600	30	120	15	90
Designation	1 k/15 s	1 k/120 s	1 k/600 s	4 k/30 s	4 k/120 s	12 k/15 s	12 k/90 s

* Varies with foam quality.

Based on standard API free fluid test (API RP 10B-2), the base slurry produced 1% free fluid during setting while all foamed slurries produced no free fluid.

Small amounts of slurries were transferred to glass vials and syringes for microstructure analysis. These samples were allowed to set before they were imaged using a 3D X-ray microscope (Model: ZEISS Xradia 520 Versa). For samples generated based on API procedures, the sample diameter ranges from 5 mm to 20 mm depending on foam quality, with corresponding CT-scan cylindrical field of view ranging from $5 \times 5 \times 5$ mm to $20 \times 20 \times 20$ mm. A standard procedure provided by the manufacture of the micro-CT was followed to obtain optimal scan parameters for each particular sample. The specific parameters, such as scan time, voltage, current, and filters used, vary depending on the sample size and density. Fig. 1 shows 2D slices of micro-CT images of a 20%-quality foamed cement scanned at different time after mixing; no detectable change in gas bubble size was found before and after cement sets. Fig. 2 shows representative 2D slices of micro-CT images of foamed cement with different foam qualities, which indicate that gas bubble size increases significantly with increasing foam quality.

In order to obtain gas bubble size distribution data, the images typically need to be smoothed and binarized. During this study, specialized 3D image analysis software (Avizo® 9.0) was used to analyze the gas bubble size distribution of the foamed cement. Basic modules of the software employed to analyze gas bubble size distribution in foamed cement include: extract region of interest (ROI), anisotropic diffusion, interactive thresholding, opening, separate objects, label analysis etc. More detailed descriptions of the application of these image processing modules can be found in [14]. The ROI are cubes and the edge/cropped bubbles are included in the analysis. This is because excluding the edge bubbles seem to increase the variations of analysis results for different ROI's of the same sample. The bubble diameter derived during this study was an equivalent diameter from the volume of the bubble (i.e. assuming a perfect sphere has the same volume as the bubble). Other image analysis software and methods may also be used to obtain gas bubble size distributions [11,12].

3. Test results and discussion

3.1. Foam quality and stability analysis

Density of the foamed slurry (ρ_{fs}) was calculated by measuring the weight of the slurry using a steel foam cement cup with a known volume, i.e.

$$\rho_{fs} = \frac{W_1 - W_2}{V} \quad (2)$$

where W_1 is the weight of the empty cup; W_2 is the weight of the cup filled with foamed cement; V is the volume of the cup. Foam quality, or gas volume fraction (ϕ_g) of the foamed slurry was then derived from the density measurements based on the following formula (API 10B-4)

$$\phi_g = \frac{\rho_{ufss} - \rho_{fs}}{\rho_{ufss}} \times 100\% \quad (3)$$

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