Petroleum and natural gas industries — Cements and materials for well cementing —

Part 4: Preparation and testing of foamed cement slurries at atmospheric pressure

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National foreword

This British Standard is the official English language version of EN ISO 10426-4:2004. It is identical with ISO 10426-4:2004.

The UK participation in its preparation was entrusted by Technical Committee PSE/17, Materials and equipment for petroleum, petrochemical and natural gas industries, to Subcommittee PSE/17/-/3, Drilling and completion fluids and well cements, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this subcommittee can be obtained on request to its secretary.

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Part 4:

Preparation and testing of foamed cement slurries at atmospheric pressure

Industrie du pétrole et du gaz naturel — Ciments et matériaux pour la cimentation des puits —

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 10426-4 was prepared by Technical Committee ISO/TC 67, *Materials, equipment and offshore structures* for petroleum, petrochemical and natural gas industries, Subcommittee SC 3, Drilling and completion fluids, and well cements.

ISO 10426 consists of the following parts, under the general title *Petroleum and natural gas industries* — *Cements and materials for well cementing*:

- Part 1: Specification
- Part 2: Testing of well cements
- Part 3: Testing of deepwater well cement formulations
- Part 4: Preparation and testing of foamed cement slurries at atmospheric pressure
- Part 5: Determination of shrinkage and expansion of well cement formulations at atmospheric pressure

Introduction

Users of this part of ISO 10426 should be aware that further or differing requirements may be needed for individual applications. This part of ISO 10426 is not intended to inhibit a vendor from offering, or the purchaser from accepting, alternative equipment or engineering solutions for the individual application. This may be particularly applicable where there is innovative or developing technology. Where an alternative is offered, the vendor should identify any variations from this International Standard and provide details.

Cements or cement blends used for foamed cement slurry preparation at atmospheric pressure should be fit for purpose. Such cements could include well cements of ISO Classes, high alumina cement, or other speciality cements. The cements and blending materials should conform to appropriate standards. Where International Standards do not exist, conformance with other appropriate standards should be made.

In this part of ISO 10426, where practical, U.S. Customary units are included in brackets for information.

Petroleum and natural gas industries — Cements and materials for well cementing —

Part 4:

Preparation and testing of foamed cement slurries at atmospheric pressure

1 Scope

This part of ISO 10426 defines the methods for the generation and testing of foamed cement slurries and their corresponding unfoamed base cement slurries at atmospheric pressure.

2 Normative references

The following normative document is indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 10426-2:2003, Petroleum and natural gas industries — Cements and materials for well cementing — Part 2: Testing of well cements

3 Sampling

3.1 General

Samples of the cement material or cement blend, solid and liquid additives, and water used for mixing are required to test a foamed cement slurry in accordance with this part of ISO 10426. Accordingly, the best available sampling technology should be employed to ensure the test materials match as closely as possible those found at the well site.

3.2 Method

Applicable sampling techniques for the fluids and materials used in foamed cementing operations can be found in ISO 10426-2:2003, Clause 4. If required, the temperatures of the mix water, cement or cement blends, and liquid additives may be measured with a thermocouple or thermometer capable of measuring temperature with an accuracy of \pm 2 °C (\pm 3,5 °F). These temperatures should be recorded. Temperature-measuring devices shall be calibrated (in the case of a thermocouple) or checked for accuracy (in the case of a thermometer) annually.

4 Slurry calculations

4.1 Calculation of base cement slurry composition with and without surfactant(s)

The final base cement slurry for preparing a foamed cement slurry contains surfactant(s), which cannot be added to the base cement slurry for initial mixing. This requires calculation of the relative mass percentage (mass fraction) of the surfactant(s) in the foamed cement slurry. This is done by taking the total mass of the

surfactant(s) and dividing by the total mass of the base cement slurry. (For these calculations, additives are considered those materials added to the cement that do not result in foaming the system.)

The mass fraction (percentage) of surfactant(s) can be calculated by:

$$w_{s} = [m_{s}/(m_{c} + m_{a} + m_{s} + m_{w})] \times 100$$
 (1)

where

 w_s is the mass fraction of surfactant(s), expressed as a percent;

 m_s is the mass of surfactant(s), expressed in grams;

 $m_{\rm C}$ is the mass of cement, expressed in grams;

 m_a is the mass of additive(s), expressed in grams;

 $m_{\rm w}$ is the mass of water, expressed in grams.

If desired, the base cement slurry density without surfactant (ρ_{bwos}) can be calculated, in grams per cubic centimetre, by:

$$\rho_{\text{bwos}} = \frac{m_{\text{c}} + m_{\text{a}} + m_{\text{w}}}{V_{\text{c}} + V_{\text{a}} + V_{\text{w}}}$$
(2)

where

 $m_{\rm c}$ is the mass of cement, expressed in grams;

 m_a is the mass of additive(s), expressed in grams;

 $m_{\rm w}$ is the mass of water, expressed in grams;

 $V_{\rm c}$ is the absolute volume of cement, expressed in cubic centimetres;

 $V_{\rm a}$ is the absolute volume of additive(s), expressed in cubic centimetres;

 $V_{\rm w}$ is the volume of water, expressed in cubic centimetres.

4.2 Determination of slurry volume and mass

4.2.1 Slurry volume

4.2.1.1 General

Determine the volume of unfoamed base cement slurry to be used. The total volume of unfoamed base cement slurry shall include the volume of surfactant(s) to be added to the base cement slurry. The surfactant(s) is(are) added after the initial mixing of the base cement slurry. The volume of unfoamed base cement slurry with surfactants to be placed in the blending container can be calculated by one of two methods (see 4.2.1.2 and 4.2.1.3).

4.2.1.2 Known gas content

When it is desired to foam a slurry with a specific volume fraction of gas per volume of slurry (foam quality), the resultant density of the foamed cement slurry must be determined. This can be calculated by:

$$\rho_{fs} = \left(\frac{100 - \varphi_g}{100}\right) \times \rho_{ufss} \tag{3}$$

where

 ho_{fs} is the density of the foamed cement slurry, expressed in kilograms per cubic metre (pounds-mass per gallon);

 $\varphi_{\rm q}$ is the volume fraction of gas in the final foamed cement slurry, expressed as a percent;

 ρ_{ufss} is the density of the unfoamed base cement slurry <u>with</u> surfactant(s), expressed in kilograms per cubic metre (pounds-mass per gallon).

4.2.1.3 Known foamed cement slurry density

When the desired density of the foamed cement slurry is known [or after calculating it with Equation (3)], determine the mass, in grams, of cement slurry including surfactant(s) to be placed into the blending container to prepare the foamed cement slurry. The mass of unfoamed base cement slurry with surfactant(s) can be calculated by:

$$m_{\rm ufss} = V_{\rm mc} \times \rho_{\rm fs}$$
 (4)

where

 $m_{\rm ufss}$ is the mass of unfoamed base cement slurry $\underline{\rm with}$ surfactant(s) to be placed in the blending container, expressed in grams;

 $V_{
m mc}$ is the blending container volume, expressed in cubic centimetres;

 ρ_{fs} is the desired density of the foamed cement slurry, expressed in grams per cubic centimetre.

4.2.2 Surfactant(s) and slurry mass

The masses of surfactant(s) and unfoamed base cement slurry required for testing are found using Equations (5) and (6).

The mass of surfactant(s) to be placed into the mixer with the unfoamed base cement slurry is determined as follows:

$$m_{\rm S} = m_{\rm ufss} \times \frac{w_{\rm S}}{100} \tag{5}$$

where

 m_s is the mass of surfactant(s), expressed in grams;

 $\it m_{ufss}$ is the mass of unfoamed base cement slurry with surfactant(s), expressed in grams;

 $w_{\rm S}$ is the mass fraction of surfactant, expressed as a percent.

The mass of base cement slurry is determined as follows:

$$m_{\rm ufs} = m_{\rm ufss} - m_{\rm s} \tag{6}$$

where

 $m_{\rm ufs}$ is the mass of unfoamed base cement slurry without surfactant(s), expressed in grams;

 $m_{\rm ufss}$ is the mass of unfoamed base cement slurry with surfactant(s), expressed in grams;

 $m_{\rm s}$ is the mass of surfactant(s) to be added to the unfoamed base cement slurry, expressed in grams.

NOTE The percentage contribution of each material by mass was determined in 4.1.

4.2.3 Additional calculations

If the density of the foamed cement slurry is known, the volume fraction (percent) of gas can be calculated by:

$$\varphi_{g} = \frac{\rho_{ufss} - \rho_{fs}}{\rho_{ufss}} \times 100 \tag{7}$$

where

 ϕ_{q} is the volume fraction of gas in final foamed cement slurry, expressed as a percent;

 ρ_{ufss} is the density of the unfoamed base cement slurry <u>with</u> surfactant(s), expressed in kilograms per cubic metre;

 ρ_{fs} is the density of the foamed cement slurry, expressed in kilograms per cubic metre.

The volume of unfoamed base cement slurry can be calculated by:

$$V_{\rm us} = V_{\rm mc} - \frac{V_{\rm mc} \times \varphi_{\rm g}}{100} \tag{8}$$

where

 $V_{\rm us}$ is the unfoamed base cement slurry volume, expressed in cubic centimetres;

 $V_{\rm mc}$ is the blending container volume, expressed in cubic centimetres;

 $\varphi_{\rm q}$ is the volume fraction of gas in final foamed cement slurry, expressed as a percent.

The mass of unfoamed base cement slurry can be calculated by:

$$m_{\rm ufss} = V_{\rm us} \times \rho_{\rm ufss}$$
 (9)

where

 $m_{\rm ufss}$ is the mass of unfoamed base cement slurry with surfactant(s), expressed in grams;

 $V_{
m us}$ is the unfoamed base cement slurry volume, expressed in cubic centimetres;

 ρ_{ufss} is the density of the unfoamed base cement slurry <u>with</u> surfactant, expressed in grams per cubic centimetre.

NOTE The density terms contained in Equations (7) and (9) can be expressed in units of kg/m³ or g/cm³.

5 Apparatus

5.1 Blending container, with a lid that seals, for preparing foamed cement slurry at atmospheric pressure in the laboratory (see Figure 1).

The blending container is similar to that used for standard slurry preparation, except it has a threaded cap with an O-ring seal. The cap has a small hole [\pm 19 mm (\pm 0,75 in) diameter] in the centre fitted with a removable plug with a vent hole. A conventional blending container that does not have a seal cannot be used for these tests.

5.2 Mixing blade assembly, either a single mixing blade as supplied by the manufacturer, or a multiple stacked-blade assembly.

Testing to date has not identified a significant difference in slurries mixed with the two different blade assemblies using the sealed blending container.

- **5.2.1** Single blade assembly, in accordance with ISO 10426-2:2003, Clause 5.
- **5.2.2 Multi-blade (stacked-blade) assembly**, constructed of a series of assemblies, each blade in accordance with ISO 10426-2:2003, Clause 5 (see Figure 1).

The assembly consists of five standard blades attached to a central shaft, and spaced equally along the shaft.



Figure 1 — Blending container and multi-blade assembly

6 Preparation of base cement slurry

6.1 Determination of blending container volume

This method assumes the base cement slurry as described in 4.1 is prepared in a separate mixing container and this prepared slurry weighed into the blending container with a sealed lid. Accurate determination of the volume of the blending container is critical to this procedure. The calculations for slurry volume, density and foamed cement slurry-to-gas ratio are based on determination of this container volume, as follows.

Weigh the clean, dry blending container (including mixing assembly, screw-on lid and screw-in plug for the lid). Remove the screw-on lid from the blending container and remove the screw-in plug from the lid. Fill the blending container with water and screw the lid on tightly. Pour additional water into the hole in the lid until the container is completely filled, and screw the plug into the lid. Wipe the excess water that exits from the plug's

vent hole and re-weigh the container. The mass of the water inside the container is then divided by the density of the water to determine an accurate volume for the blending container.

The volume of the blending container should be checked any time the blades are replaced, or after any damage to the container that may affect the volume. The volume should be verified at least every 6 months.

NOTE Preparation of sufficient volume of the base cement slurry may require multiple mixes using the standard mixing procedure, or use of a large laboratory blender. See preparation of large slurry volumes in ISO 10426-2:2003, Annex A.

6.2 Base cement slurry preparation

6.2.1 General

Base slurries containing all additives except foaming surfactant(s) shall be prepared in accordance with ISO 10426-2:2003, Clause 6.

6.2.2 Temperature considerations

If possible, the temperatures of the cement sample, additives and mix water should be within \pm 2 °C (\pm 3,5 °F) of the respective temperatures recorded or anticipated at surface at the well site. (This is NOT the anticipated temperature in the well, but at surface conditions at the well site.) The temperature of the blending container should approximate that of the mix water being used in the slurry design. The blending container assembly shall be calibrated annually to rotate at tolerances of \pm 200 r/min at 4 000 r/min and \pm 500 r/min at 12 000 r/min.

6.2.3 Density measurement

The density of the unfoamed base cement slurry can be determined by methods found in ISO 10426-2:2003, Clause 6.

7 Preparation of foamed cement slurry at atmospheric pressure

7.1 General

Based on the mass calculated in 4.2, weigh the appropriate amount of the prepared base slurry into the blending container. Add the calculated amount of surfactant(s). The final mass of the base cement slurry and added surfactant(s) should be checked against the final desired base cement slurry mass calculated in 4.2.1.3.

7.2 Generation of a foamed cement slurry

Place the lid and plug on the container and make sure the blending container is sealed. Using the blade assembly described in either 5.2.1 or 5.2.2, mix the slurry at the 12 000 r/min setting for 15 s.

Because of the increase in slurry volume and viscosity, the maximum revolutions per minute of the blending container blade(s) may be less than 12 000 r/min. The maximum attainable revolutions per minute depend on the power of the blending container motor, slurry density and foam quality. Record and report the final revolutions per minute of the mixer blade(s).

During the mixing, there will be a noticeable change in the sound (pitch) from the blending container. After mixing, there may be some slight pressure in the blending container, due to temperature increases and energy imparted to the foam during the foaming process. Care shall be exercised when removing the top of the blending container. After mixing, open the sampling port or container lid, and check that the slurry completely fills the blending container.

If the slurry does not fill the blending container at the end of the 15 s period, it is doubtful the slurry will foam properly under field conditions. The slurry should be redesigned.

When preparing the foamed cement slurry in the sealed blending container, it is common for the final density of the foamed cement slurry to be less than designed. This is the result of pressure-generated expansion due to the mixing energy, and due to the relaxation of the surfactant which produces an increase in relative bubble size. The foamed cement slurry may expand upon removal from the blending container. One method to obtain a foamed cement slurry having a density closer to the design density is as follows:

- a) design the cement slurry density to be foamed, for example: 1 893 kg/m³ base cement slurry foamed to 1 318 kg/m³ (15,8 lbm/gal foamed to 11,0 lbm/gal);
- b) prepare the 1 318 kg/m³ (11,0 lbm/gal) foamed cement slurry in the laboratory according to the design;
- c) measure the density of the foamed cement slurry, e.g. 1 246 kg/m³ (10,4 lbm/gal);
- d) if the measured density is less than the design, check design calculations;
- e) if the calculations are correct, subtract the measured density from the design density to obtain an "offset correction" e.g. $1.318 \text{ kg/m}^3 1.246 \text{ kg/m}^3 = 72 \text{ kg/m}^3$ offset correction (11,0 lbm/gal 10,4 lbm/gal = 0,6 lbm/gal offset correction);
- f) recalculate the slurry density using the offset correction, e.g. $1.318 \text{ kg/m}^3 + 72 \text{ kg/m}^3 = 1.390 \text{ kg/m}^3$ (11,0 lbm/gal + 0,6 lbm/gal = 11,6 lbm/gal);
- g) prepare a new foamed cement slurry according to the corrected density, e.g. 1 893 kg/m³ foamed to 1 390 kg/m³ (15.8 lbm/gal foamed to 11.6 lbm/gal);
- h) measure the density of the foamed cement slurry; the density of this foamed cement slurry should be close to the desired density of 1 318 kg/m³ (11,0 lbm/gal);
- i) if this density is still not acceptable, obtain a new offset correction and prepare a new base cement slurry;
- j) if the measured foam density is above the design, it will be difficult to obtain the proper foamed cement density in the field, and the slurry should be redesigned.

8 Example calculations for the preparation of foamed cement slurry at atmospheric pressure

8.1 General

The following calculations demonstrate the use of the equations in 4.1 and 4.2 to determine the proper quantities of a base cement slurry and surfactant.

Problem: foaming a base cement slurry of density 1 737 kg/m³ (14,5 lbm/gal) with a 31 % volume fraction of gas.

Slurry design: Cement + 0,017 75 m³/tonne surfactant (Cement + 0,2 gallons/sack surfactant)

Base cement slurry density = 1.737 kg/m^3 (14,5 lbm/gal) Surfactant density = 1.198 kg/m^3 (10 lbm/gal)

Desired volume fraction of gas = 31 % Container volume = 1 170 cm³

NOTE One tonne equals 1000 kg.

8.2 Mass percentage calculations

The relative mass percentage contribution for the surfactant(s) is calculated. (Calculations in parentheses are based on U.S. per sack mass, and are not meant to be equivalent to the metric values.)

	Mass	Volume	(Mass)	(Volume)
Cement	1 000 kg	0,318 7 m ³	(94 lbm)	(3,59 gal)
0,017 75 m ³ /tonne surfactant	21,3 kg	0,017 75 m ³	(2 lbm)	(0,2 gal)
Water	590 kg	0,590 m ³	(55,39 lbm)	(6,65 gal)
Total	1 611,3 kg	0,926 45 m ³	(151,39 lbm)	(10,44 gal)

Calculation of mass fraction (percent) contributions:

Cement	$(1\ 000\ kg/1\ 611\ kg) \times 100 = 62,1\%$	$[(94 \text{ lbm}/151,39 \text{ lbm}) \times 100 = 62,1\%]$
Surfactant	$(21,3 \text{ kg/1 611 kg}) \times 100 = 1,3\%$	[(2 lbm/151,39 lbm) \times 100 = 1,3%]
Water	$(590 \text{ kg/1 611 kg}) \times 100 = 36,6\%$	$[(55,39 \text{ lbm}/151,39 \text{ lbm}) \times 100 = 36,6\%]$

8.3 Calculation of slurry density without surfactant(s)

The density of the base cement slurry without surfactant ($\rho_{\rm bwos}$) is calculated by:

	Mass	Volume	(Mass)	(Volume)
Cement	1 000 kg	0,3187 m ³	(94 lbm)	(3,59 gal)
Water	590 kg	0,590 m ³	(55,39 lbm)	(6,65 gal)
Total	1 590 kg	0,9087 m ³	(149,39 lbm)	(10,24 gal)

$$\rho_{bwos} = 1\ 590\ kg/0,9087\ m^3 = 1\ 749\ kg/m^3 \quad \text{(149,39 lbm/10,24 gal} = 14,60\ lbm/gal)$$

8.4 Calculation of foamed cement slurry density with known volume fraction of gas

For example, using Equation (3) in 4.2.1.2:

$$\begin{split} \rho_{\rm fs} & = [(100-\varphi_{\rm g})\,/\,100]\times 1\,\,749\,\,{\rm kg/m^3} = [(100-31)\,/\,\,100]\times 1\,\,749\,\,{\rm kg/m^3} \\ \\ \rho_{\rm fs} & = 1\,\,207\,\,{\rm kg/m^3} \quad (=1,207\,\,{\rm g/cm^3}) \end{split}$$

8.5 Calculation of required grams of unfoamed base cement slurry

For example, using Equation (4) in 4.2.1.3:

$$m_{\text{ufss}} = 1 \ 170 \ \text{cm}^3 \times 1,207 \ \text{g/cm}^3$$

 $m_{\text{ufss}} = 1 \ 412,2 \ \text{g}$

8.6 Calculation of required grams of surfactant and slurry

For example, using Equation (5) in 4.2.2:

$$m_{\rm S} = 1.412,2 \times (1,3 / 100)$$

 $m_{\rm S} = 18,36 \text{ g}$

For example, using Equation (6) in 4.2.2:

$$m_{\text{ufs}} = 1 \, 412.2 \, \text{g} - 18.36 \, \text{g}$$

 $m_{\text{ufs}} = 1 \, 393.8 \, \text{g}$

8.7 Summary of example calculations

To prepare a foamed cement slurry sample from the example slurry in a 1 170 cm³ container requires:

1 393,8 g of base cement slurry;

18,36 g of surfactant.

9 Atmospheric testing of foamed cement slurries

9.1 General

Because of the high volume of gas in a foamed cement slurry, it is necessary to modify some of the standard testing procedures to prevent erroneous test results.

9.2 Determination of foamed cement slurry density

The density of the foamed cement slurry shall be determined by pouring the foamed cement slurry into a container with a large open top that has a known volume when completely filled. Weigh the container, pour the foamed cement slurry into the container and level the top with a straight blade. Wipe the outside of the container clean and again weigh the container with the slurry. The density of the foamed cement slurry in the container is determined by dividing the slurry mass by the container volume and converting to the appropriate density units.

A pressurized fluid density balance should never be used to determine the density of a foamed cement slurry prepared at atmospheric pressure, since this can compress the gas bubbles and the slurry density indication will be too high. A non-pressurized slurry density balance is not recommended, because the small hole in the centre of the lid can cause a restriction, resulting in partial pressurization of the slurry. This can cause errors in the density determination.

9.3 Determination of foamed cement slurry stability

9.3.1 Stability of unset foamed cement slurry

Evaluate the foam stability by pouring a sample of the foamed cement slurry into a standard 250 ml graduated cylinder, or other appropriately sized container. Seal the top of the cylinder to prevent dehydration. Place the cylinder on a stable, vibration-free counter-top and let stand for a 2 h period. Periodically examine the slurry during the 2 h period. The purpose of this test is to check for settling and stability in the foamed cement slurry, and to record the visual appearance of the foamed cement slurry (e.g. free fluid, settling, bubbles concentrated in specific area, etc.). The cylinder contents cannot be cured at temperatures above ambient because an increase in temperature will increase the bubble size and slurry volume, and may affect the slurry stability.

Density measurements may be made of the foam at multiple locations in the cylinder after the 2 h period if desired. To determine the density of the slurry at various locations in the cylinder, use a large syringe with a flexible tube attached to remove small portions from the top, middle and bottom. (Use of a catheter or irrigation-type syringe is recommended.) The removed slurry can be transferred to a smaller graduated cylinder to determine the mass of a known volume of the slurry. The density can then be determined.

9.3.2 Stability of set foamed cement slurry

Check the foamed cement slurry stability by curing samples until they are set, and determine the density gradient throughout the sample.

Samples may be cured in non-greased, covered cylinders 50,8 mm in diameter \times 101,6 mm in height (2 in \times 4 in) or any appropriate covered container. Use of grease or other mould-release agents should be avoided, as these materials can affect the stability of the foamed cement slurry.

Allow the slurry to cure for 24 h, or until set. Remove the cement from the tube. The length of the set cement specimen should be measured. Mark the specimen into at least three segments of approximately equal length. Cut the sample into sections and mark them from the top to the bottom. The specimen should not be cut with a saw that uses water, since the specimen can absorb water and the density of the specimen may change. Large variations in density from sample top to bottom are an indication of instability.

Determine the mass of each section in air and in water as follows. Place a beaker of fresh water on a balance and tare the balance to zero. Place a section on the balance beside the beaker. Record the mass and remove the section from the balance. Tare the balance to zero. Place a noose of thin line around the section. Pick up the section by the line and suspend the section in the water in the beaker such that the sample is totally immersed in water and does not touch the bottom or sides of the beaker. Obtain the mass of the sample immersed in water as quickly as possible to prevent excessive water absorption. Remove the sample from the water. Repeat the procedure for each set cement section. By applying the Archimedes Principle, calculate the density of each cement sample by:

$$\rho_{\rm S} = \frac{m_{\rm a}}{m_{\rm W}} \tag{10}$$

where

 $\rho_{\rm s}$ is the density of the sample, in grams per cubic centimetre;

 m_a is the mass of the sample in air, expressed in grams;

 $m_{\rm w}$ is the mass of the sample in water, expressed in grams.

9.3.3 Evaluating foamed cement slurry stability at temperature < 90 °C (194 °F)

Prepare an appropriate mould for curing the foamed cement slurry sample. For example, a PVC curing mould can be prepared by applying primer/cleaner and glue to the PVC parts and assembling them (Figure 2). Allow sufficient time for the glue to harden. Apply sealing tape to the brass fittings. (PVC or other plastic material is preferred because the foamed cement slurry will not bond to the mould material. Other materials are acceptable provided a mould-release compound is not used on the surface of the mould.)

Pour the foamed cement slurry into the mould and screw the large brass (or other corrosion-resistant material) fitting into the top. Slurry must exit the centre hole of the large brass fitting. Then screw the small brass plug into the large brass fitting and tighten both. Cure at the desired temperature until set. The specimen may be cured in a vertical position or at a specific angle if desired.

After curing, cool to room temperature, remove the brass fitting and plug from the top, and examine the specimen. Note any obvious problems in the top of the specimen. Cut the PVC mould into at least 3 pieces, marking each piece to refer to the relative position (i.e. top, middle and bottom). The specimen should not be cut with a saw that uses water, since water can be absorbed by the specimen and change its density. Carefully cut the PVC longitudinally along each segment until the PVC can be removed. Examine the set foamed sections for signs of instability. The specimens can then be tested for density using the Archimedes Principle described above. Compressive strength can also be determined on each section, providing the sample is a uniform cylinder and the ends are smooth and planar. The length-to-diameter ratio, as well as the use of cubes vs. cylinders, can have a dramatic effect on the determined strength.

NOTE Specimen geometry affects the values determined for compressive strength, i.e. strength determined with one specimen geometry may not correspond to that of a different geometry.

9.3.4 Signs of foam instability

Signs of instability include

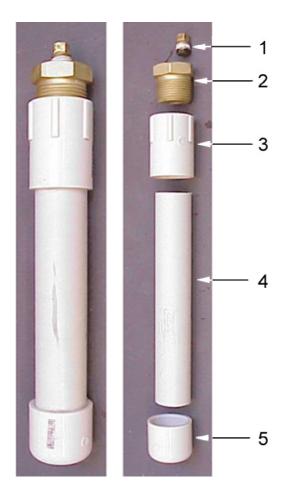
- more than a trace of free fluid,
- bubble breakout noted by large bubbles on the top of the sample,
- excessive gap at the top of the specimen (minor meniscus effects are normal),

- visual signs of density segregation as indicated by streaking or light to dark colour change from top to bottom,
- large variations in density from sample top to bottom.

9.4 Determination of compressive strength

Pour the foamed cement slurry into a curing mould that can be sealed. (The sealing lid prevents the foamed cement slurry from expanding as it is heated. The expansion can result in an undesired density decrease.) A suitable mould is a standard 50,8 mm (2 in) cube mould (described in ASTM C $109^{[1]}$) with a cover (without grooves) and gasket clamped on. Plastic cylindrical moulds, 50,8 mm in diameter \times 101,6 mm in height (2 in \times 4 in) with a sealable top have also been used. The length-to-diameter ratio, as well as the use of cubes, as compared to cylinders, can have a dramatic effect on the determined strength. The sample ends shall be parallel, smooth and planar.

Place the sealed mould containing the foamed cement slurry into an atmospheric-pressure water bath, cure the specimen and determine the compressive strength in accordance with ISO 10426-2:2003, 7.5.3.



Key

- 1 6,35 mm (1/4 in) brass plug
- 2 25,4 mm x 6,35 mm (1 in x 1/4 in) brass reducer
- 3 25,4 mm (1 in) PVC collar
- 4 25,4 mm (1 in) PVC schedule 40 tubing, length 152 mm to 203 mm (6 in to 8 in)
- 5 25,4 mm (1 in) PVC cap

Figure 2 — Example of curing mould for evaluation of foamed cement slurry stability

9.5 Determination of permeability

For determination of the permeability of foamed cement slurry, pour the foamed cement slurry into permeability test moulds and cure in the mould. Exercise care to prevent damage to the specimen if the foamed cement slurry is poured into a mould from which the cured specimen must be removed, or cored, cut or sealed in the testing apparatus. Curing should be conducted under atmospheric pressure, as for the determination of compressive strengths.

Perform permeability testing of the cured specimens in accordance with ISO 10426-2:2003, Clause 11.

10 Determination of other properties of base unfoamed cement slurry

10.1 General

A slurry foamed at atmospheric pressure shall not be tested under pressure. Applying pressure to a foamed cement slurry prepared at atmospheric pressure will compress the foam, changing the density and gas ratio. This can also allow contamination when tested for thickening time in a high pressure-high temperature (HPHT) consistometer.

For the following tests, prepare the unfoamed base cement slurry without the surfactant(s) in accordance with ISO 10426-2:2003, Clause 5. After the slurry is prepared, stop the mixer, add the surfactant(s) and stir gently with a spatula to distribute it uniformly in the slurry. It is recommended the slurry be transferred gently from the blending container to a beaker and back three times to ensure a uniform distribution of the surfactant(s). The use of a small amount of a material intended for preventing/breaking air entrainment in slurries that are not foamed is permitted for these tests.

10.2 Determination of thickening time

As surfactant(s) can affect the thickening time, the thickening-time test is normally performed using a standard HPHT consistometer on the unfoamed base cement slurry containing the surfactant(s).

Perform the thickening-time test on the unfoamed base cement slurry in accordance with ISO 10426-2:2003, Clause 9.

10.3 Determination of fluid loss

Fluid-loss tests performed on a foamed cement slurry prepared at atmospheric pressure may not yield reliable results. The fluid-loss values obtained from a foamed cement slurry are lower than those from a base unfoamed cement slurry. The fluid loss of the unfoamed base cement is normally used as an indication of the fluid loss of the foamed cement slurry.

Perform the static fluid loss test on the unfoamed base cement slurry containing the surfactant(s) in accordance with ISO 10426-2:2003, Clause 10.

10.4 Determination of rheological properties

Use of a rotational viscometer with a foamed cement slurry may result in separation of the gas from the slurry, causing erroneous results. A correlation can be used to convert the rheological properties of the unfoamed base slurry to that of a foamed cement slurry with varying foam qualities to simulate the field conditions.

Perform the rheological test on the unfoamed base cement slurry containing the surfactant(s) in accordance with ISO 10426-2:2003, Clause 12.

Bibliography

[1] ASTM C 109, Standard test method for compressive strength of hydraulic cement mortars (using 2-in or [50-mm] cube specimens)

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