

From Pickering foams to porous carbonate materials: crack-free structuring in drying ceramics



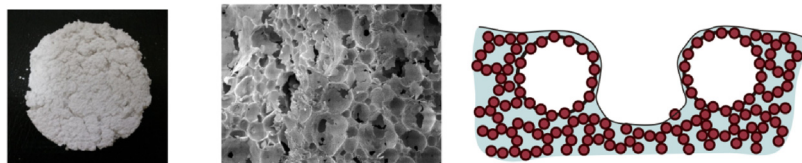
M. Hristova^a, I. Lesov^a, S. Tcholakova^{a,*}, V. Goletto^b, N. Denkov^a

^a Department of Chemical and Pharmaceutical Engineering, Faculty of Chemistry and Pharmacy, Sofia University, 1 J. Bourchier Ave., 1164 Sofia, Bulgaria

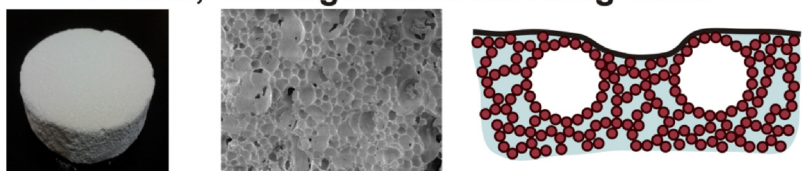
^b Saint Gobain Recherche, 39 quai Lucien Lefranc, 93303 Aubervilliers, France

GRAPHICAL ABSTRACT

SLES, crack formation



C10Ac, homogeneous receding front



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ABSTRACT

Particle-stabilized foams have attracted considerable research interest, due to their long term stability (months to years) and the possibility to use them as precursors for production of porous materials with hierarchical porosity. In our previous study [Lesov et al., *J. Colloid Interface Sci.* 504 (2017) 48–57] we clarified the role of the rheological properties of the foamed suspensions and the type of foam film stabilization in the production of porous silica materials with low mass density and excellent insulating properties. In the current study we extend our approach to prepare lightweight carbonate ceramics with controlled density, shrinkage and good mechanical properties. To prepare the wet foam precursors, we tested a series of eight anionic surfactants which were previously reported to provide sufficient hydrophobization of CaCO_3 particles and long-term stability of the liquid foams. From those surfactants, the medium-chain fatty acids led to crack-free porous materials with superior mechanical strength, compared to the conventional surfactants. We study the reasons for the formation of cracks in drying Pickering foams and, on this basis, propose optimal conditions for obtaining dry porous carbonate materials with required porosity. Mechanistic explanations are proposed for the main observed effects.

* Corresponding author.

E-mail addresses: mk@lcpe.uni-sofia.bg (M. Hristova), lesov@lcpe.uni-sofia.bg (I. Lesov), SC@LCPE.UNI-SOFIA.BG (S. Tcholakova), valerie.goletto@saint-gobain.com (V. Goletto), nd@lcpe.uni-sofia.bg (N. Denkov).

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1. Introduction

Porous ceramics is a class of materials which is usually characterized with low mass density and high specific surface area. These properties make the porous ceramics desirable materials for vast range of applications, spanning from simple construction blocks to highly efficient catalyst supports, and even scaffolds for tissue engineering. Advantageous method for their preparation is the direct foaming technique, in which a concentrated particle suspension is foamed, dried and often sintered [1–3].

Gonzenbach et al. [4] showed that choosing appropriate surfactant and tuning its concentration, boosts the foaming of suspensions of hydrophilic particles. One recommendation for selection of appropriate surfactant in terms of foam stability is to use surfactants with an electrical charge opposite to that of the particles [4,5]. Such oppositely charged surfactants adsorb on the particles surface and modify *in situ* the particle hydrophobicity during foaming [4]. Some moderate particle hydrophobicity is considered as a prerequisite for particle adsorption on the surface of the foam bubbles and for ensuring the desired long-term stability of the formed Pickering foams [5–23].

Studies related to flotation industry, generation of hydrophobic coatings and stabilization of Pickering foams revealed that several anionic surfactants are suitable for (partial) hydrophobization of carbonate particles [24–30]. Thus, Somasundaran and Agar [24] showed that sodium dodecyl sulfate (SDS) is appropriate for the modification and flotation of calcite particles. Hana and Anazia [25–27] used carboxylic acids and their salts for similar purpose. Later on, other authors used oleic acid [28,29], dioctyl sodium sulfosuccinate (AOT) [30], and alkylbenzene sulfonic acid [31].

Zhou et al. [29] used the knowledge on carbonate modification to prepare Pickering foams in presence of oleic acid. The obtained wet foams were stable to coalescence and Ostwald ripening but suffered from water drainage, due to the relatively low concentration of carbonate particles and the respective low yield stress of the suspension. Similar results were obtained by Cui et al. [30], who used SDS and AOT for the surface modification of carbonate particles. However, the foams in [30] had low air fraction (e.g. below 50 vol. %), also contained particles with low concentrations and suffered from water drainage which made them inappropriate precursors for preparation of light-weight porous materials [32–34].

In our previous studies [33,34], we established that a well-defined, optimal ratio between the particle and surfactant concentrations may ensure very stable foam precursors. On one side, the surfactant content should be sufficiently high to partially hydrophobize the particles and to trigger their adsorption on the bubble surface during foaming. The particle hydrophobization should provide also moderate attraction between the neighboring particles to induce an optimal yield stress of the

foamed suspensions which can stabilize the foams to drainage [20,33,34]. On the other hand, the surfactant concentration had to be sufficiently low to avoid the severe particle aggregation into large clusters and strong gels which would suppress the suspension foamability [33,34] and cause cracking in the drying foams [33–35].

The major aim of the current study is to apply the approach from [33,34] and to prepare carbonate ceramic materials with controlled porosity and optimal mechanical strength by selecting the most appropriate type and concentration of surfactant in the foamed carbonate suspensions. To achieve this aim we compare the effects of eight anionic surfactants, most of which have been reported previously to provide sufficient hydrophobization of CaCO₃. We found that particularly appropriate for formation of dry porous materials of high quality are the medium-chain fatty acids with 8 to 10 carbon atoms. The reasons for the formation of severe cracks in the drying carbonate foams are studied and explained mechanistically.

2. Materials and methods

2.1. Materials

We used CaCO₃ particles, provided as dry commercial powder (Omyabrite® 1300 X – OM, Omyacorp), containing 98 wt% calcium carbonate, 1 wt% humidity and 1 wt% insoluble salts. The average size of the particles provided by the manufacturer is 2.4 μm and the BET surface area is 25–30 m²/g (ISO 9277). The mass density of the particles is 2700 kg/m³.

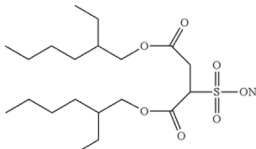
The anionic surfactants, listed in Table 1, were used to modify the particles' surface and to assist the suspension foaming. The conventional surfactants (viz. all except the fatty acids) were first prepared as 10 wt% stock solutions, while the fatty acids were used as received. Decanoic and dodecanoic acid were melted first (1 min at 50–60 °C) and then added to the suspensions.

All suspensions and surfactant solutions were prepared with deionized water, produced by Elix 3 module (Millipore, USA).

2.2. Suspension preparation

Suspensions with 30 wt% particle concentration were prepared in the following way: 180 g CaCO₃ particles were measured in a polyethylene jar and 420 g deionized water was added. This mixture was hand shaken and then placed on a rotating mill (BML-2, Witeg) for 10 min at 100 rpm. Afterwards, the suspension was homogenized for 3 × 10 min with a pulse sonicator (SKL1500-IIDN, Ningbo haishu sklon developer). The latter was set to 1 s long pulses with power output of 1200 W, followed by 0.5 s off, using a sonotrode with diameter of 20 mm. The suspension was then left to cool down to room temperature

Table 1
List of surfactants studied.

Name	In text	Chemical Formula	Producer	Average molecular weight, g/mol	Active content, %
Sodium α-olephin sulfonate	α-OS	(C _n H _{2n-1})SO ₃ Na (n ≈ 15)	AAKO	314.0	90
Sodium dodecyl sulfate	SDS	CH ₃ (CH ₂) ₁₁ SO ₄ Na	Acros	288.4	99
Linear alkylbenzene sulfonate	LAS	C ₆ H ₅ CH(C _n H _{2n+1})SO ₃ Na (n ≈ 11.8)	Sigma	348.5	90
Dioctyl sodium sulfosuccinate	AOT		Sigma-Aldrich	444.6	99
Sodium lauryl ether sulfate	SLES	C ₁₂ H ₂₅ (OCH ₂ CH ₂) ₃ SO ₄ Na	Stepan Co.USA	420.5	70
Octanoic acid	C8Ac	C ₇ H ₁₃ COOH	Fluka	144.2	99
Decanoic acid	C10Ac	C ₁₀ H ₂₁ COOH	Alfa Aesar	172.3	98
Dodecanoic acid	C12Ac	C ₁₃ H ₂₇ COOH	Acros	200.3	99

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