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RESEARCH ARTICLE OPEN ACCESS

Shear-crystallization in beeswax oleogels: structural modulation and insights

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Abstract – Oleogels are increasingly recognized as promising materials for food and cosmetic applications, yet their industrial production still faces major challenges due to limited understanding of cooling treatments. This study presents the first systematic comparison of static versus shear-crystallization in coconut–sunflower oil oleogels structured with beeswax (7.5–12.5% w/w) at 20 °C and 30 °C. Texture and rheological analyses confirmed that static-crystallized oleogels exhibited significantly higher firmness and yield viscosity, while shear-crystallized samples formed softer, shear-thinning gels with lower yield viscosity. However, polarized light microscopy revealed that static crystallization promoted the formation of unwanted large granular crystals (>100 μm), attributed to post-crystallization of high-melting fractions during storage. In contrast, shear-induced crystallization produced finer, more uniform crystal networks (3.0–4.6 μm). These findings highlight shear-crystallization as a novel and effective approach for regulating crystal growth in beeswax oleogels, offering controlled pathways to improve stability and overall quality for industrial food and cosmetic products.

Keywords: oleogel / beeswax / coconut oil / shear-crystallization

Résumé – **Cristallisation sous cisaillement dans les oléogels à la cire d'abeille : modulation de la structure et nouveaux éclairages.** Les oléogels sont de plus en plus reconnus comme des matériaux prometteurs pour les applications alimentaires et cosmétiques. Toutefois, leur production à l'échelle industrielle reste confrontée à d'importants défis, en raison d'une compréhension encore limitée des traitements de refroidissement. Cette étude présente la première comparaison systématique entre la cristallisation statique et la cristallisation sous cisaillement dans des oléogels à base d'un mélange d'huiles de coco et de tournesol, structurés par de la cire d'abeille (7,5–12,5 % m/m), à 20 °C et 30 °C. Les analyses de texture et les mesures rhéologiques ont confirmé que les oléogels obtenus par cristallisation statique présentaient une fermeté et une viscosité de seuil significativement plus élevées, tandis que les échantillons cristallisés sous cisaillement formaient des gels plus souples, rhéofluidifiants, avec une viscosité de seuil plus faible. Cependant, les observations en microscopie à lumière polarisée ont montré que la cristallisation statique favorisait la formation de gros cristaux granulaires indésirables (> 100 μm), attribués à une post-cristallisation des fractions à haut point de fusion au cours du stockage. À l'inverse, la cristallisation induite par cisaillement produisait des réseaux cristallins plus fins et plus homogènes (3,0–4,6 μm). Ces résultats mettent en évidence la cristallisation sous cisaillement comme une approche innovante et efficace pour contrôler la croissance des cristaux dans les oléogels à la cire d'abeille, ouvrant des perspectives pour améliorer la stabilité et la qualité globale des produits alimentaires et cosmétiques à l'échelle industrielle.

Mots clé : Oléogel / cire d'abeille / huile de coco / cristallisation sous cisaillement

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Highlights:

- Oleogels showed shear-thinning behavior and yield viscosity rose with beeswax content.
- Static crystallization produced firmer gels but heterogeneous structures prone to large crystal growth.
- Shear-crystallization formed uniform fine crystals and weaker gels that strengthened during 2–4 weeks of storage.
- Stronger shear produced smaller crystals, improved homogeneity, and enhanced stability.

1 Introduction

Oleogels are innovative structured fat systems formed by entrapping liquid oils within a solid three-dimensional network (Martins *et al.*, 2020; Manzoor *et al.*, 2022; Huang *et al.*, 2023). Typically, the oil phase consists of edible vegetable oils, while the structuring agents include proteins, polysaccharides, or fat crystals (Qiu *et al.*, 2018; Winkler-Moser *et al.*, 2019; Silva *et al.*, 2022). Among the various oleogelators, waxes are the most widely employed due to their low toxicity, reasonable cost, and high crystallization capacity. Over the past decade, numerous oleogel systems have been successfully developed using candelilla wax, beeswax, sunflower wax, and rice bran wax (Thakur *et al.*, 2023; Frolova *et al.*, 2022; Doan *et al.*, 2018).

Oleogels exhibit unique rheological properties by combining both liquid-like and solid-like behaviors, making them promising candidates for diverse applications in the food and cosmetic industries (Liu *et al.*, 2024; Pawar *et al.*, 2024). In food systems, their high content of unsaturated fatty acids allows oleogels to serve as healthier fat alternatives in products such as margarine, sausages, and chocolate spreads (Silva *et al.*, 2021; Ozer *et al.*, 2023; Zhang *et al.*, 2023; Tirgarian *et al.*, 2023). Moreover, diacylglycerol oil-based oleogels have recently been incorporated into peanut butter formulations to reduce phase separation and improve fat stability during storage (Ding *et al.*, 2024). In cosmetics, oleogels are increasingly used in skin-care formulations, where they enhance moisture retention, slow down skin aging, and act as carriers for bioactive compounds (Yaqoob *et al.*, 2024; Yan *et al.*, 2025; Smeu *et al.*, 2025; Hanifah *et al.*, 2018).

Despite these advantages, large-scale industrial production of oleogel remains challenging. Conventional preparation methods include static crystallization of oil mixtures (direct method) and freeze-drying of oil-in-water emulsions (indirect method) (Doan *et al.*, 2018). The indirect method is limited by homogenization and dehydration efficiency, while the direct method often produces non-uniform structures (Liu *et al.*, 2024). Besides, common industrial fat-based products such as margarine and shortening typically employ shear-induced crystallization to enhance structural homogeneity (Nguyen *et al.*, 2020), rather than relying on static crystallization. Recent investigations into monoacylglyceride oleogels have shown that strong shear can accelerate polymorphic transitions of fat crystals (Rondou *et al.*, 2025). Similarly, shear-crystallization has been reported to produce smaller crystals and greater plastic dissipation in oleogel microstructures compared to static crystallization (Werner-Carcamo *et al.*, 2023). Nevertheless, the mechanistic understanding of shear-crystallization in oleogels remains limited, with most studies conducted on small sample volumes.

To address these gaps, the present study investigated the effects of shear versus static crystallization on the structural and rheological properties of beeswax-structured oleogels prepared from coconut–sunflower oil blends, which are common ingredients in cosmetic formulations. Crystallization was performed at 20 °C and 30 °C using a benchtop crystallizer, and the resulting oleogels were characterized through texture analysis, rheometry, and polarized light microscopy to elucidate how processing conditions influence microstructure and stability.

2 Materials and methods

2.1 Materials

Coconut oil and sunflower oil were purchased from a local supplier. Beeswax (BW) was provided by Cosman Chemical (China). The major fatty acids in BW included palmitic acid (14.02%), n-docosane (2.03%), linoleic acid (1.41%), 2-methyloctacosane (16.54%), stearic acid (9.40%) and pentacosane (56.43%).

2.2 Sample preparation

The formulation of oleogels was summarized in Table 1 and Figure 1. Briefly, sunflower and coconut oils were mixed together with a ratio of 1:1 (w/w) at 45 °C for 10 minutes while beeswax was completely melted at 60 °C (according to the supplier's guidelines). Then melted BW was added to vegetable oils at different concentrations (7.5–12.5%, w/w) (Sivakanthan *et al.*, 2024; Werner-Carcamo *et al.*, 2025). For static crystallization (SC), the mixtures were slowly cooled at 30 °C and 20 °C (using incubator and cooling chambers).

For shear-crystallization (SH), 1000 mL mixtures were cooled to 30 and 20 °C at rotational speeds of 30 rpm and 45 rpm using a benchtop crystallizer (LR1000, IKA, Germany) equipped with an anchor stirrer. The cooling was conducted by using a circulating refrigerator bath (TC150-MX, Brookfield, USA).

All samples were placed into plastic cups (40 mm in diameter), each containing 35 g, and stored at 30 °C and 20 °C for 28 days.

2.3 Texture analysis

For large deformation analysis, the firmness of oleogel was determined by a texture analyser (TA.XT, Stable Microsystem, UK) equipped with a cylinder probe (diameter of 30 mm) using the compression mode. The penetration depth was 10 mm (50% of sample height) and the test speed was 1 mm/s. The data was obtained by software Exponent 6.0 (Stable Micro-system, UK). Measurement tests were conducted after 2 hours, 2 days, 7 days, 14 days and 28 days of storage.

2.4 Rheological analysis

For small deformation analysis, viscosity profiles of oleogel were determined using a rheometer (DVT2-EXTRA, Brookfield, USA) equipped with a vane spindle (V-73).

Table 1. Oleogels formulation

Sample	Treatment	Beeswax (%)	Vegetable oils (%)
SC-7.5	Static crystallization	7.5	92.5
SC-10	Static crystallization	10.0	90.0
SC-12.5	Static crystallization	12.5	87.5
SH30-7.5	Shear-crystallization at 30 rpm	7.5	92.5
SH30-10	Shear-crystallization at 30 rpm	10.0	90.0
SH30-12.5	Shear-crystallization at 30 rpm	12.5	87.5
SH45-7.5	Shear-crystallization at 45 rpm	7.5	92.5
SH45-10	Shear-crystallization at 45 rpm	10.0	90.0
SH45-12.5	Shear-crystallization at 45 rpm	12.5	87.5

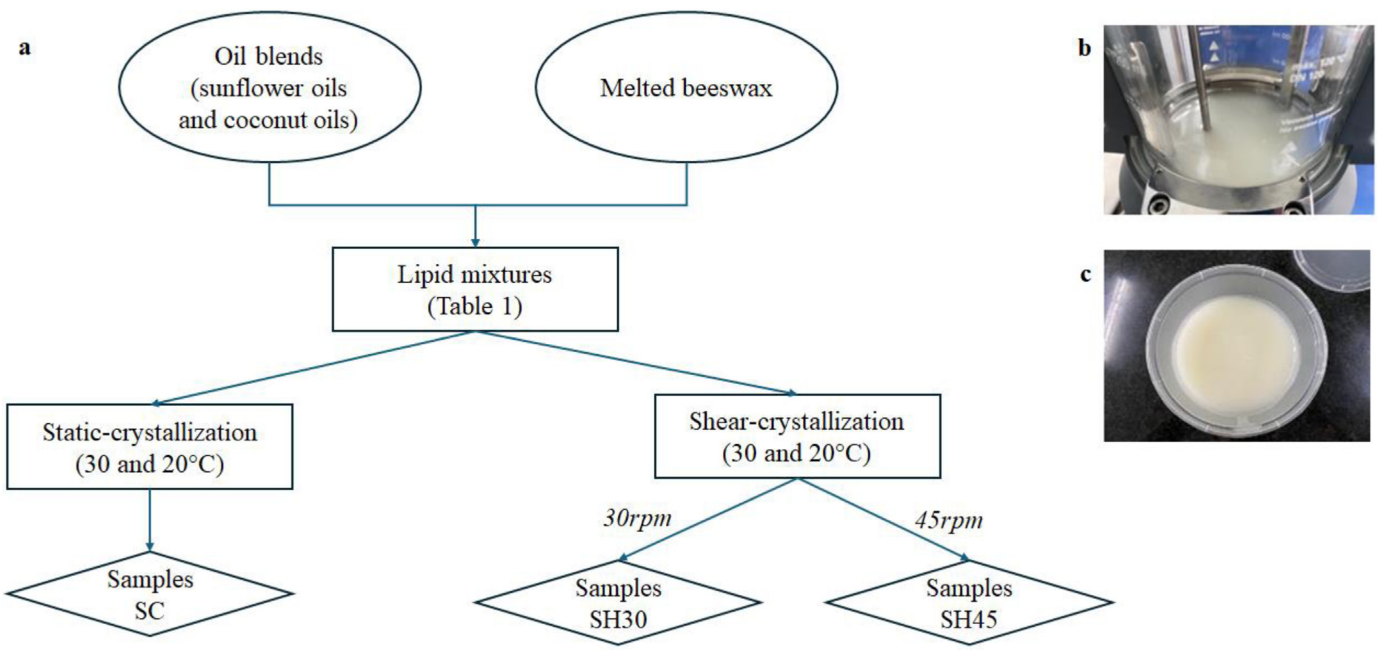

Fig. 1. Oleogel formulations (a: schematic diagram, b: benchtop crystallizer, c: oleogel).

Measurements were performed across a rotational speed range of 1–200 rpm, and data acquisition was carried out using RheocalcT 1.0 software (Brookfield, USA). All tests were conducted after 28 days of storage.

2.5 Polarization microscopy

The morphology of fat crystals in oleogels was examined using a polarized light microscope (DM2500P, Leica, Germany) equipped with a digital camera (DFC450, Leica, Germany). Images were captured using LAS v4.6 software (Leica, Germany), and crystal particle size was quantified with ImageJ software (NIH, USA). Measurement tests were conducted after 2 days, 14 days and 28 days of storage.

2.6 Statistical analysis

All measurements were conducted triplicated. Two-way ANOVA and Least Significant Difference (LSD) were applied to evaluate the effect of beeswax content and storage time on the textural properties of oleogel. Statistical analysis was conducted using software SPSS (v16, IBM, USA) with a significant difference of 0.05.

3 Results and discussion

3.1 Oleogel formation

The influence of beeswax concentration and crystallization process on oleogel formation is presented in Figure 2. In static-crystallized samples, oleogels were successfully formed at 30 °C only when the oleogelator concentration exceeded 10% (samples SC-10 and SC-12.5). At lower beeswax concentrations, the crystal network was insufficient to immobilize the liquid phase (Han *et al.*, 2022). When the crystallization temperature was reduced to 20 °C, oleogel formation was observed even in sample SC-7.5, as partial crystallization of coconut oil near room temperature contributed to strengthening the microstructure (Thomas *et al.*, 2023; Dhulipalla *et al.*, 2023). In contrast, the

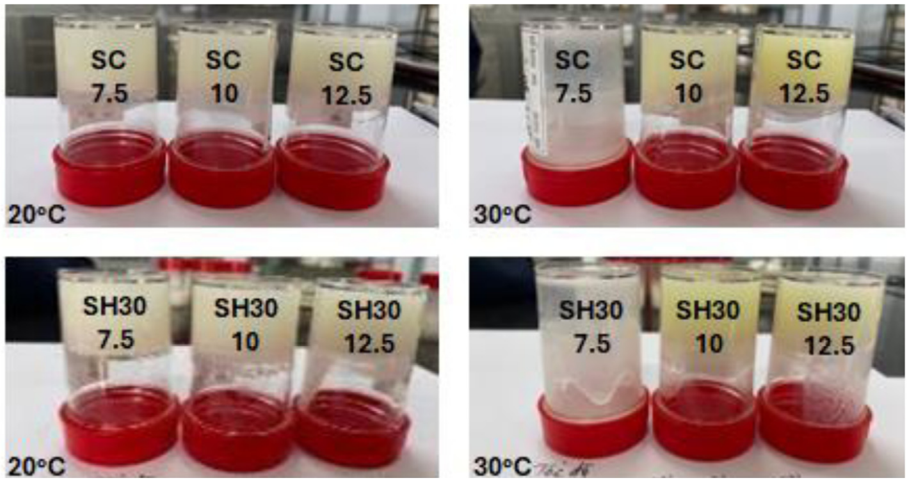

Fig. 2. Static-(SC) and shear-crystallized (SH30) oleogels (7.5, 10 and 12.5% BW) at different temperatures (20 and 30 °C).

behavior of shear-crystallized samples was more complex. At both investigated temperatures, SH samples remained in the liquid phase (weak gels) immediately after crystallization (Fig. 1) and the transition mainly occurred during storage accompanying with the slow increase of gel firmness (Fig. 3). Similar delayed solidification has been reported in previous studies on shear-crystallization of vegetable oil blends (Nguyen *et al.*, 2020, 2021). This phenomenon can be explained by the effect of shear flow, which suppresses crystal agglomeration. Consequently, the development of a continuous crystal network occurs progressively during storage through sintering between dispersed fat crystals.

3.2 Texture and rheological properties

The effect of oleogelator concentration and crystallization method on the texture properties of oleogels is summarized in Figure 3. Firstly, the firmness of static-crystallized samples was higher than those of shear-crystallized samples, consistent with previous reports on dynamic crystallization of vegetable oils (Doan *et al.*, 2018; Nguyen *et al.*, 2020, 2021). In the absence of shear flow (SC samples), fat crystals of beeswax and coconut oil could be formed at 30 °C and 20 °C, respectively, creating a rigid shell that entrapped the remaining liquid oil (such as sunflower oil). Consequently, the mechanical resistance of these samples was strongly influenced by crystallization temperature and oleogelator concentration. Higher beeswax content resulted in greater firmness, while lower crystallization temperatures also enhanced firmness due to the crystallization of coconut fat below 25 °C (Silva *et al.*, 2022).

By contrast, SH30 and SH45 samples exhibited lower firmness and weaker gel networks. Agitation during cooling hindered crystal growth, resulting in less developed structures. However, their crystalline networks appeared more homogeneous compared with SC samples. For instance, structural collapse was frequently observed when pressing SC oleogels, whereas SH30 and SH45 maintained smoother gel structures (Supplementary S1). Notably, post-crystallization was evident in SH samples but not clearly in SC oleogels. As shown in Figure 3, the firmness of SH30 and SH45 increased during storage at both tested temperatures, with this trend persisting for two to four weeks depending on storage conditions.

Besides large-deformation test, small-deformation rheological analysis is also conducted, and the results are summarized in Figure 4. The data revealed that beeswax oleogels are shear-thinning materials at both 20 and 30 °C. While static-crystallized samples (SC) exhibited a hard gel structure characterized by high yield viscosity, shear-crystallized oleogels (SH) displayed soft gel structures with lower yield viscosity. Consistent with the texture profile analysis, samples with higher beeswax concentrations demonstrated increased yield viscosity. In addition, all samples showed elevated yield stress when crystallized at lower temperatures, attributable to the crystallization of coconut fat between 20 and 30 °C. Agitation speed also exerted a considerable influence on oleogel microstructure. Although shear-crystallized samples generally formed weak gels, SH45 samples exhibited higher yield stress compared with SH30 samples at both investigated temperatures. This difference suggests that agitation intensity plays a role in network development. In static-crystallized oleogels, crystal network formation is primarily associated with crystal growth through heterogeneous nucleation. By contrast, the structural development of shear-crystallized oleogels appears to be governed by post-crystallization processes (Huang *et al.*, 2024). In industrial fat-based products, tiny crystals typically form during cooling, and their subsequent aggregation entraps the liquid phase to generate weak gel structures. At higher agitation speeds during shear-crystallization, smaller fat crystals are produced, which promote more effective sintering and yield a stronger solid network during storage.

3.3 Crystal growth

Polarized light microscopy (PLM) was employed to elucidate the microstructure of fat-based oleogels, with representative images shown in Figures 5 and 6. As expected, SC samples exhibited low structural homogeneity. At 30 °C, fat crystal formation was primarily driven by beeswax crystallization (Winkler-Moser *et al.*, 2019; Tran *et al.*, 2023), and the number of crystals increased with higher oleogelator content.

Fig. 3. Firmness of static- (SC) and shear-crystallized (SH) oleogels (7.5, 10, 12.5% BW) during storage at different temperatures (20 and 30 °C). Values followed by different letters (a, b, c, d and e) indicate significant differences among sample means during storage ($p < 0.05$). Values followed by different numbers ([1, 2] and [3]) indicate significant differences ($p < 0.05$) among sample means with varying beeswax (BW) content.

At this temperature, most solid particles displayed a needle-like morphology (4.7–5.9 µm). However, at 20 °C, these crystals tended to aggregate into bundles and clusters (7.9–9.8 µm). This transformation was likely associated with the post-crystallization of coconut fat (Silva *et al.*, 2022). When storage temperature was reduced below the melting point, coconut oil triacylglycerols were absorbed into pre-existing beeswax crystals, leading to heterogeneous crystallization

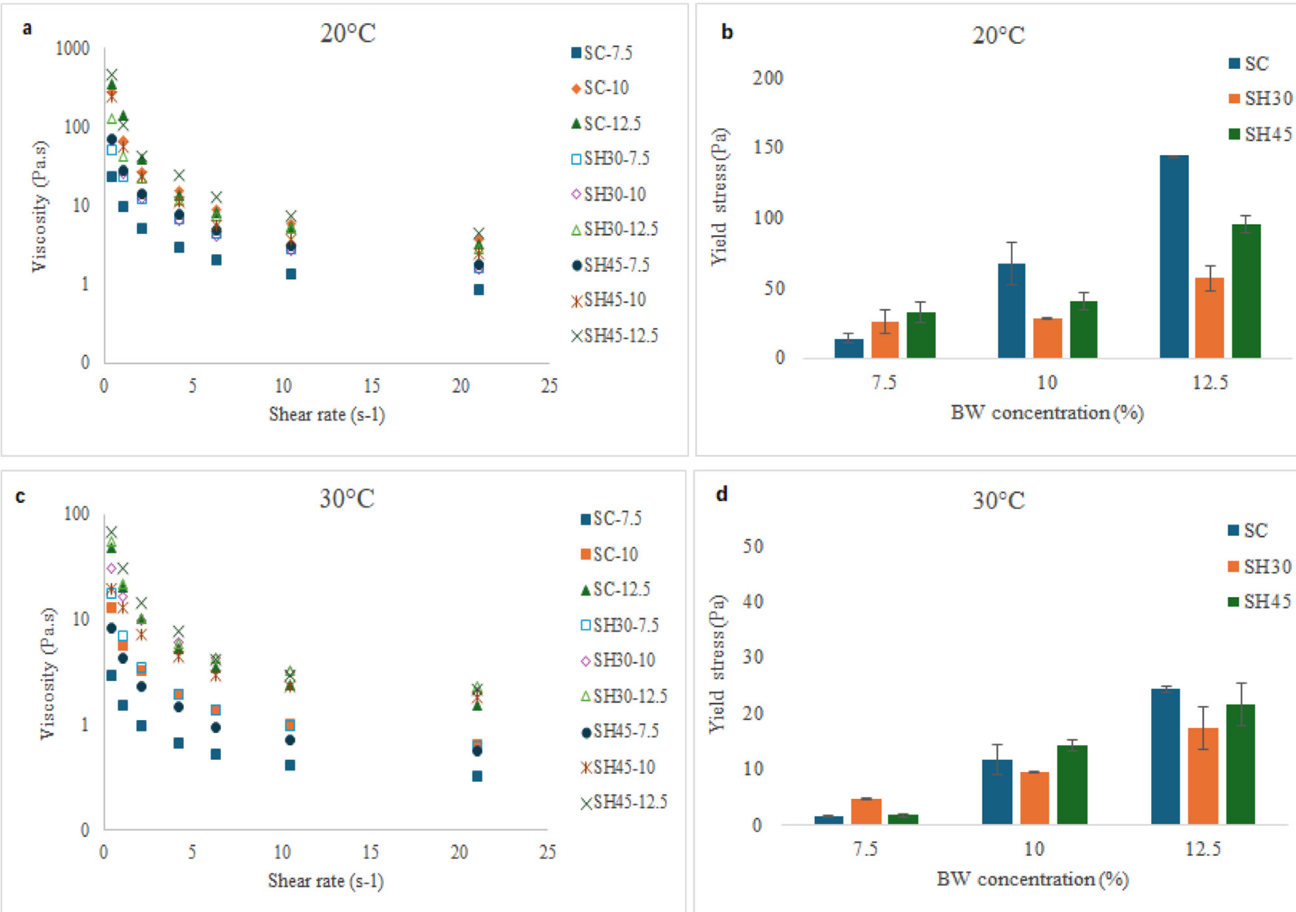

Fig. 4. Rheological properties of static- (SC) and shear-crystallized (SH) oleogels (7.5, 10 and 12.5% BW) at different temperatures (20 and 30 °C).

and gradual growth of mixed crystals. After prolonged storage, unwanted spherulites (11.96–13.04 μm) and even large granular crystals (>100 μm) were observed in sample SC (Fig. 7), which may contribute to undesirable roughness or grainy texture in products.

In contrast, SH oleogels displayed more uniform microstructures, with only minor differences between samples. Most solid particles were tiny needle-like crystals (3.0–4.6 μm), and their abundance increased with beeswax concentration. During storage, crystal agglomeration could partly occur to form clusters (6.6–9.4 μm) but the aggregation was significantly limited, and large granular crystals were absent. Unlike the post-crystallization observed in SC samples, the phase transition of coconut oil in SH samples was effectively controlled by shear-crystallization. Under strong shear flow, triacylglycerols lacked sufficient time to aggregate, while beeswax and coconut fat crystals were continuously disrupted during cooling. This process promoted the formation of fine crystals and enhanced oleogel homogeneity. Because crystal growth was well-regulated during shear-crystallization, subsequent structural changes during storage were primarily driven by sintering between solid particles rather than agglomeration. Consequently, SH oleogels maintained a more consistent and stable microstructure during long-term storage compared with SC samples.

4 Conclusion

This is the first study to systematically compare static and shear-crystallization in beeswax-structured coconut–sunflower oil oleogels. As hypothesized, static crystallization produced oleogels with high initial firmness and yield viscosity, yet their heterogeneous microstructure and tendency to form unwanted large granular crystals during storage compromised stability. In contrast, shear-crystallization generated finer, more uniform crystals (3.0–4.6 μm), reduced agglomeration, and improved homogeneity. Although shear-crystallized oleogels initially exhibited weaker gel strength, their networks progressively strengthened during storage through sintering, with firmness increasing two- to threefold depending on storage temperature. These results demonstrated that shear-crystallization could offer a long-term stability and provide practical guidance for tailoring oleogel properties to enhance texture and shelf-life in food and cosmetic products. Future work may further explore their applications in producing novel shortenings, margarine, or lotion creams.

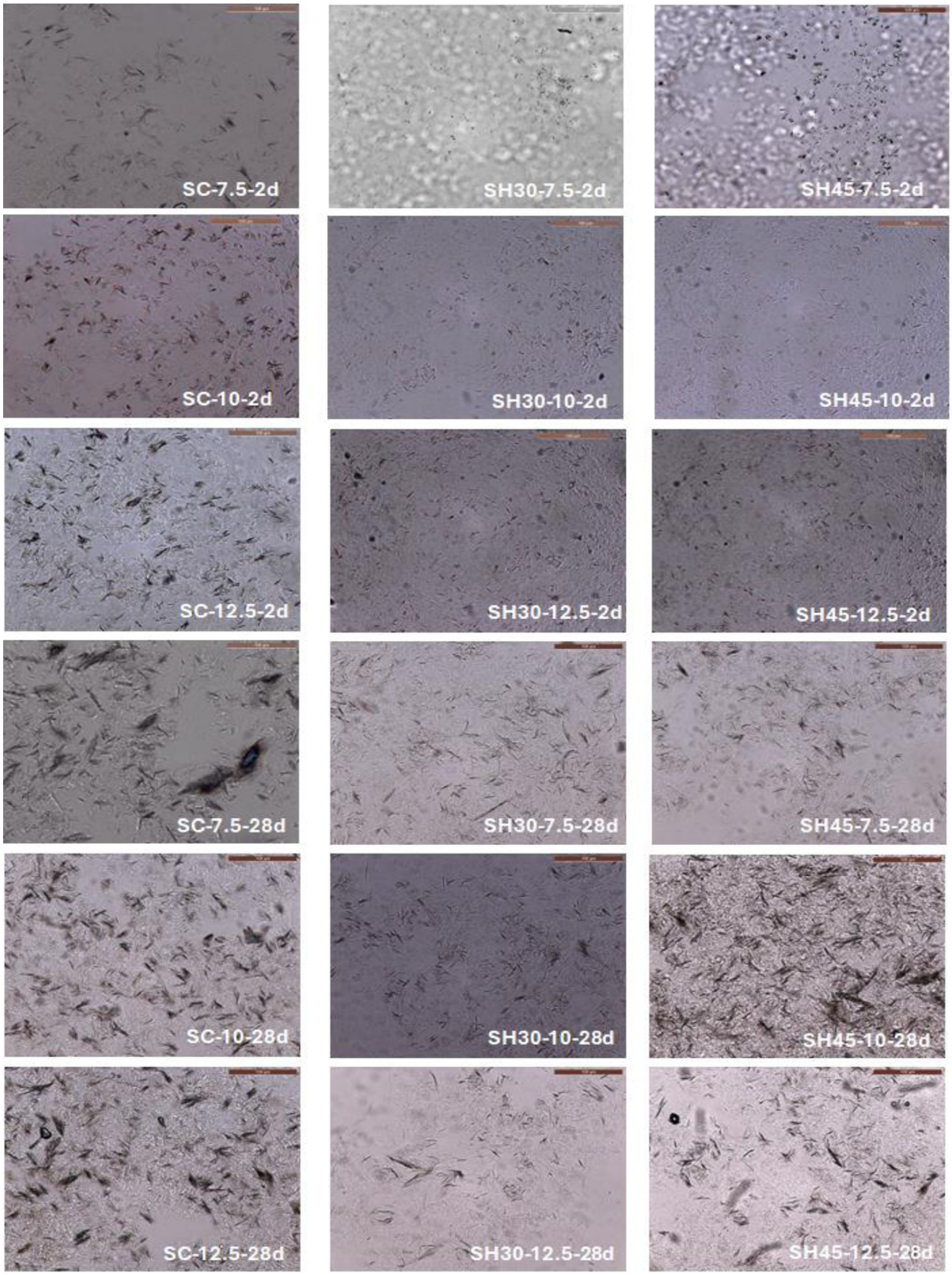

Fig. 5. Microstructure of static- (SC) and shear-crystallized (SH) oleogels (7.5, 10 and 12.5% BW) at 30 °C. Scale bar: 100 μm.

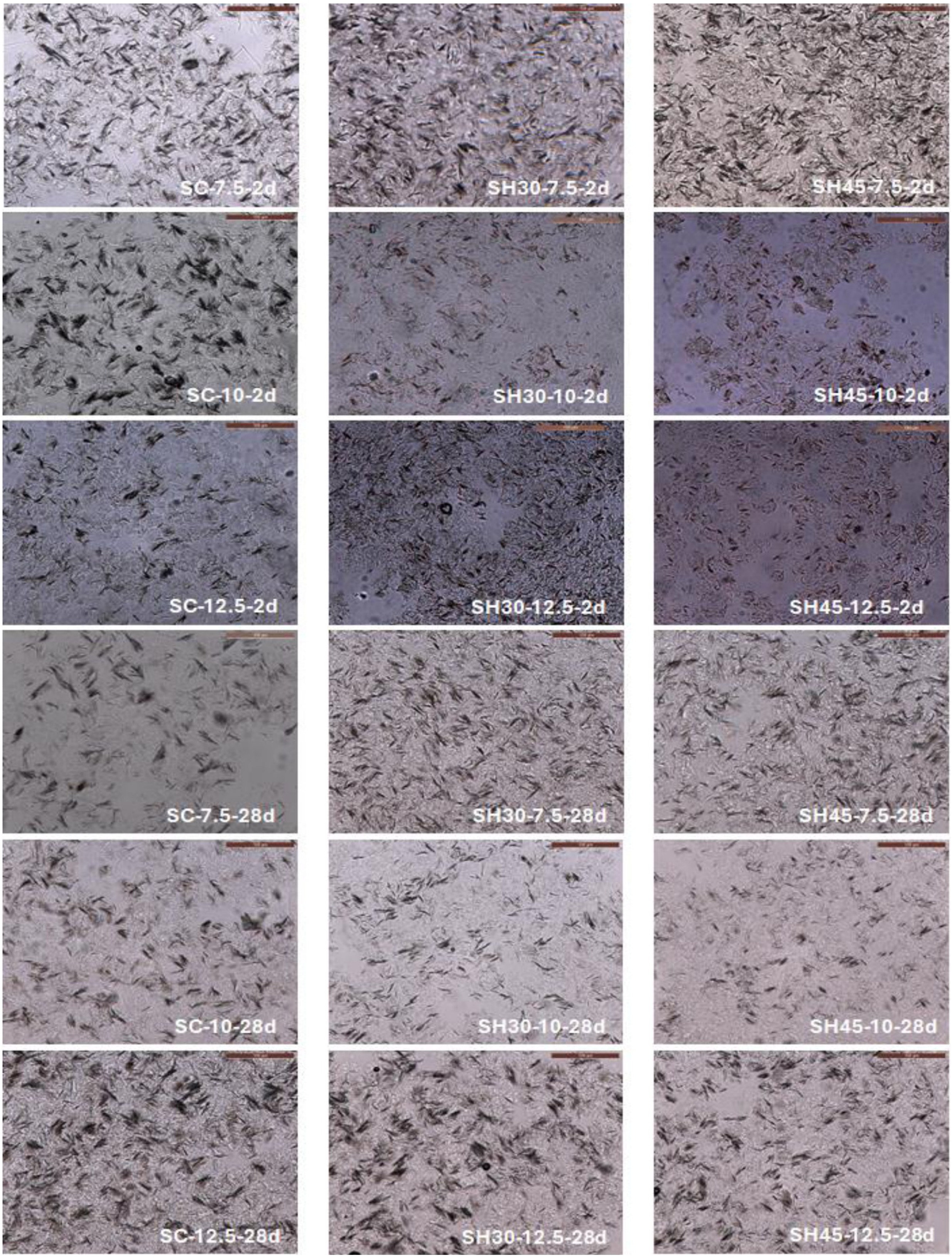

Fig. 6. Microstructure of static- (SC) and shear-crystallized (SH) oleogels (7.5, 10 and 12.5% BW) at 20 °C. Scale bar: 100 μm.

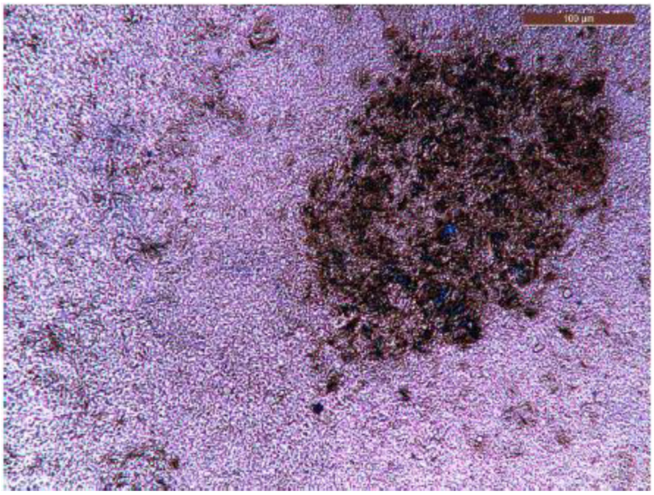

Fig. 7. Granular crystals in static-crystallized sample after one month storage. Scale bar: 100 μm.

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Conflicts of interest

There are no conflicts to declare.

Author contribution statement

Thao T. P. Tran: Investigation, data curation, writing – original draft; Phuong T. Nguyen: Investigation, data curation; Viet Nguyen: Supervision, conceptualization, writing – review editing.

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