Rheology of milled black cumin
(Nigella sativa L.)

Abstract Black cumin (Nigella sativa L.) paste is a local food in the Middle East, which is produced from dehulled black cumin seeds. The rheological properties of a commercial black cumin paste in the temperature range of 5–40 °C have been studied. Shear-thinning flow behavior with a yield stress was observed for the samples tested. The Herschel-Bulkley model described well the flow curves of the black cumin pastes at different temperatures. It was found that the yield stress, the consistency coefficient, and the flow behavior index decrease with temperature. As far as the effect of steady shearing on the rheological properties of black cumin paste is concerned, the structural kinetic approach was used to predict the thixotropic behavior. It was found that the thixotropic behavior of black cumin paste increases with increasing shear rate and is mitigated by increasing temperature. The completely destructed black cumin paste flow curves were also measured after subjecting the samples to a high shear rate for 2 h. The flow curves of the completely destructed samples were modeled well by the Herschel-Bulkley model. A comparison between the fresh and the completely destructed black cumin paste results at different temperatures revealed that there was a systematic difference, such that the completely destructed paste showed lower yield stress and flow behavior index, and showed a greater consistency coefficient.

Keywords Rheology · Black cumin · Herschel-Bulkley model · Thixotropy · Nigella sativa L.

Introduction

Nigella sativa L., also known, as black cumin is a member of the Ranunculaceae family and native to some parts of the Mediterranean region (Babayan et al. 1978). The seeds are used by Egyptians as a diuretic, carminative, and flavoring agent, by the Syrians for cheese flavoring, and by the Armenians for flavoring cheese and bakery products. On the other hand, the oil expressed from the seeds is sold in the markets of some of Middle East countries as a topical treatment for pain and stiffness in the joints. It has also been shown that seed extracts inhibit the growth of Escherichia coli, Bacillus subtilis, and Streptococcus fecalis (Saxena and Vyas 1986). On the other hand, Hanafy and Hatem (1991) found that the extract of N. sativa seeds showed antibacterial synergism with streptomycin and gentamicin and showed additive antibacterial action with spectinomycin.

Black cumin seeds are of a high nutritive value as it is rich in fat (35.5–37.8 wt%), protein (21–23.2 wt%), carbohydrates (34.4–35.0 wt%), and some minerals, such as calcium (100 mg/100 g), phosphorus (807–840 mg/100 g), and iron (9 mg/100 g). The seeds also
contain moisture (4.9–5.5 wt%) and ash (3.7–4.1 wt%). These figures were adapted from different sources (Nergiz and Ötles 1993; Abu-Jadayil et al. 1999).

Black cumin paste is a local food in the Middle East, which is produced from dehulled black cumin seeds. Black cumin paste is the product of the milled seeds of black cumin, which were de-hulled and roasted without adding or removing any of its constituents.

In Jordan there are many commercial plants, some of which are equipped with modern production lines and are automated, producing black cumin paste. A major concern facing the black cumin industry is the production and maintenance of the product while preserving the proper consistency, stability, color, and texture properties. Reliable and accurate rheological characterization of foodstuffs is necessary for the control of quality, texture, and shelf life, and for the design of flow processes, and to evaluate heating and cooling rate during different engineering operations (Dimonte et al. 1998).

The rheological properties of black cumin paste, to the best of the author's knowledge, were not examined. The seeds and oil of N. sativa have been subjected to a range of pharmacological (e.g., Salomi et al. 1992; Chakravarty 1993; Houghton et al. 1995) and chemical (e.g., Babayan et al. 1978; Nergiz and Ötles 1993; Abu-Jadayil et al. 1999) investigations in recent years. Three commercially cultivated black cumin seed varieties of Turkish origins were analyzed, and the characteristics and constituents of the seed oils were reported by Ustun et al. (1990). Dandik and Aksoy (1996) investigated the possible applications of the native lipase of black cumin seed in oleochemical reactions in relation to the process parameters. The investigated enzyme-catalyzed reactions were the hydrolysis of used frying oil and esterification of oleic acid with glycerol and methanol. Turkay et al. (1996) investigated the decarboxylation of high-acidity oils from black cumin seeds with supercritical carbon dioxide at two temperatures (40 and 60 °C), pressures (15 and 20 MPa), and polarities (pure CO₂ and CO₂/10% MeOH). An empirical kinetic model for the extraction of black cumin seed oil with supercritical carbon dioxide as solvent was developed by Fullana et al. (1999). Differential extraction yields were correlated with the factors that affected the rate (particle size, degree of milling, fluid flow rate, pressure, temperature, moisture, and time). Abu-Jadayil et al. (1999) investigated the bioavailability of iron from Jordanian plants (black cumin seeds, milk thistle seeds, sesame seeds, and thyme leaves). They found that iron was better utilized from black cumin seeds as indicated by liver storage of iron.

This work aims to investigate the steady rheological properties (i.e., the shear stress as a function of shear rate) of commercial black cumin paste and to develop methods for characterizing the time-dependent rheological behavior of black cumin paste under the effect of different temperatures and shearing conditions.

### Experimental setup and methodology

#### Materials and setup

Black cumin samples were obtained from a local company in Jordan (Al-Yarmouk Co., Amman, Jordan). Rheological properties of black cumin paste were measured with a concentric cylinder Haake-VT 500 viscometer, which has an inner cylinder rotating in a stationary outer cylinder. Three bobs (MV1, MV2, and MV3) with lengths of 60 mm and radii of 20.0, 18.4, and 15.2 mm and a cup with a radius of 21.0 mm were used to form gap widths of 1.0, 2.6, and 5.8 mm, respectively. The viscometer was thermostatically controlled with a water circulator (Haake D8) at the desired temperature with a precision of ±0.1 °C.

#### Methodology

The experiments performed to characterize the shear-, time-, and shear history dependency of the flow behavior of black cumin paste consisted of a series of three measurements:

1. **Flow curves measurements:** a fresh sample was loaded into the annular gap of the concentric cylinder viscometer. Samples were left to reach the desired temperature. The flow curves of black cumin paste were measured in the temperature range of 5.0–43.0 °C by increasing (forward measurements) and decreasing (backward measurements) the shear rate. The period of the experiment was 8 min and the values of the shear rate and shear stress were recorded every 0.5 min. The shear rate was varied from 2.20 to 131.9 s⁻¹.

2. **Shear stress measurements as a function of time at constant shear rate:** in transient measurements a fresh sample was sheared at constant shear rates, namely at 2.20, 19.93, and 47.43 s⁻¹, and the shear stress was measured as a function of shearing time until an equilibrium state was reached. The procedure was then repeated with other fresh samples at other shear rates.

3. **Completely destructed black cumin paste flow curves:** measurements of the flow curves of the completely destructed black cumin samples were conducted on samples which had been subjected to a long preshearing period to ensure that the material reached a completely destructured state at which the rheological behavior is no longer dependent on shearing time. Based on the transient measurements, it was found that preshearing the black cumin paste at shear rate of 47.43 s⁻¹ in the viscometer for 2 h was sufficient to break down the material structure and remove the thixotropic effects.

In order to investigate the reproducibility of the results, two replicates were made for most of the experiments and the reproducibility was ±3% on average.

### Results and discussion

#### Flow curves

It should first be pointed out that no surface slip was observed in the viscometer system used. Figure 1 shows the apparent viscosity of black cumin paste as a function of shear rate measured with different systems, which have different gap widths. It is clear that the apparent viscosity of black cumin paste is independent of the measuring system. Since the slip conditions encountered in a viscometer are function of the gap width, the data points presented in Fig. 1 show that the slip conditions in our system are not clear.