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Evaluation of the mechanical properties of compacted paraffin powders. Effect of formulation

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ABSTRACT

The mechanical characteristics of a paraffin–vegetable oil material and the compressive behavior of the powder stemming from this material were used to estimate the resistance of the compressed samples. The compressive behavior of powders under the low pressure range (1–2 MPa) applied in the candle industry was investigated in order to predict the tensile strength of the compressed samples. Compressive behavior was evaluated under lab conditions similar to those practiced in the candle industry. Compressive behavior of the powders, which represents the resistance of the particles to rearrangement during the packing step, K_p (30.98 ± 1.20 kPa of the mixture M1), was positively correlated to tensile strength of the compressed samples, σ_t (175.46 ± 3.61 kPa of mixture M1). Tensile strength of the compacts was also related to the mechanical properties of the raw material: high tensile strength was linked to low ductility (γ_{MR}), high mechanical strength (R_{MR}) and high Young's modulus (E) of the material. Formulation—particularly the presence of a lubricant of mineral (0.52%) and vegetable (44.1%) origin in mixture M5—was found to strongly influence the mechanical properties of the compressed samples ($\sigma_t = 115.52 \pm 2.42$ kPa).

1. Introduction

The paraffin wax obtained from refining oil finds a wide range of applications in industry, medicine, and food, and is the main material used in compression-based candle manufacture. However, despite being in widespread use, the behavior of paraffin as a powder in compression has never yet been studied. Furthermore, research into alternative renewable materials of vegetable origin is thriving as the candle market looks for more sustainable sources. Several patents [1–3] state that the combination of alternative materials with paraffin wax has to meet the physical characteristics required, including controlled melting points, high malleability, low fragility, and high chemical stability. Vegetable oils of different origins (e.g. palm oil, rapeseed oil and olive oil) are candidates for mixing with paraffin, and the use of vegetable oils derived from vegetable waste is also described in certain patents [4]. In order to incorporate alternative materials in the candle, one has to verify their compatibility with a complex mechanical process, i.e. compression, to guarantee the quality of the final candle product.

The compression process is widely used in many industrial sectors, from pharmacy and metallurgy to cosmetics and foods. The success of the compression is related to the mechanical properties of the material [5–6] and of the powder bed, i.e. the ability of the powder to decrease in volume under pressure (compressibility) [7–11] and form a cohesive compact by densification (compactibility) [12–13], the transmission of forces through the powder volume [14–15], the mechanism involved in the cohesion of the tablet (solid bridges, forces of attraction, entanglements) [16–17], and inter-particle/wall friction during the compression [18–20]. All these parameters play an important role in the formation of a cohesive compressed sample.

The mechanical properties of the materials themselves, such as the ductile or brittle character of the material and its hardness/softness characterized by Young's modulus [21–26], also have to be taken into account. These parameters depend on the nature of the forces involved and the structure and purity of the material.

The behavior of the powder in compression is critical in the formation of the compressed sample. During compression, with the increase of the pressure, the powder undergoes intensive densification and the powder particles move together to generate cohesive forces [17,19,27]. Many studies have focused on the relationship between relative density of the powder and compaction pressure, considered as defining the compressibility of a material, and several models have been developed and modified to characterize the compressibility of powders [7–11,28]. When compression starts, the particles rearrange themselves by sliding and rotating to form a denser stack, thus

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increasing the number of connection points between the particles [7–8,10–11]. With the increase in pressure, at the end of the packing step the particles cannot slide further and undergo deformations according to their mechanical characteristics [7–8,17,29–33]. The specific energy required during compression is related to the densification of the powder and depends mainly on pressure range and the physical properties of the material [34–36].

The tensile (or breaking) strength of compressed samples depends on the nature and intensity of the inter-particle bonds, and the contact surface between the particles. Comparisons of various materials are generally based on destructive tests [37–42]. Many studies have been carried out to understand the mechanisms of rupture of the compressed samples and to define the influence of various parameters on material strength. Several studies have investigated the relationship between tensile strength and the properties of the compressed samples, including compaction behavior, porosity, particle size, and particle shape [43–50]. Concerning the shape and surface of the grains, the more the grains are rough and irregularly shaped, the stronger the forces of inter-particle friction, which promotes the formation of solid bridges and improves the strength of the compressed samples [43–44,49].

Another factor contributing to the mechanical strength of the compressed sample is the presence of lubricant. The role of lubricants used in the manufacture of pharmaceutical tablets is to reduce the friction of the matrix walls and avoid powder sticking to the punches, and to improve the transmission of pressures in the volume of the powder bed by reducing the friction between the particles [51–55]. Although these parameters can improve the compression characteristics, they can also have negative effects on the quality of the grains, and especially on the mechanical strength of the samples [56–61].

The objective of this study was to provide a thorough understanding of the formation of a cohesive compacted sample from paraffin/vegetable substitute powders, and to define the predominant factors that explain the tensile strength of the compressed samples. The study was performed in three steps: (i) study of the mechanical characteristics of the material (deformation capacity and mechanical strength of the material, Young's modulus); (ii) study of the formation of the compressed samples (the compressive behavior of the powders) and energy requirements; (iii) determination of the tensile strength of the compressed samples. Particular emphasis was placed on the step of packing under low pressure, where rearrangement of the particles is the dominant behavior. The influence of formulation (paraffin and vegetable substitute) and presence of lubricant in the formulation was studied in the three characterization steps.

The main purpose of this study was to produce compressed samples with the highest tensile strength while working in low pressure ranges (up to 2 MPa) used in candle manufacture.

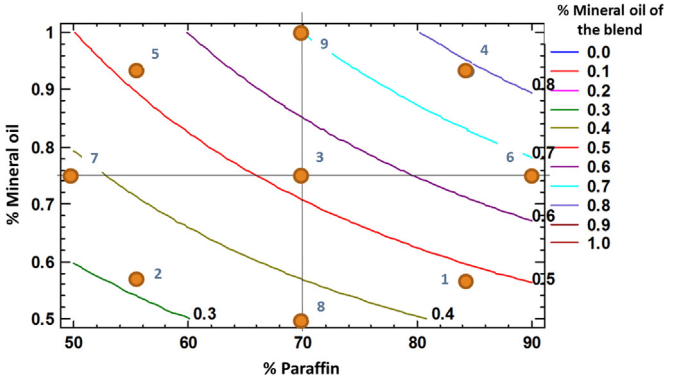

Fig. 1. Experimental area of the second-order central composite design.

Table 1
Composition and characteristics of the five mixtures. Mean ± standard deviation.

Mixture	Composition			Melting point (°C)	Density (g/cm^3)
	% Paraffin (fully and semi)	% Vegetable oil	% Mineral oil of the blend		
M1	84.1	15.9	0.48	54.27 ± 0.23	0.92 ± 0.002
M2	55.9	44.1	0.32	49.43 ± 0.41	0.93 ± 0.002
M3	70	30	0.53	48.10 ± 0.15	0.92 ± 0.002
M4	84.1	15.9	0.78	52.45 ± 0.32	0.91 ± 0.001
M5	55.9	44.1	0.52	49.56 ± 0.67	0.93 ± 0.003

2. Materials and methods

2.1. Materials

Two types of paraffin and a transformed vegetable oil (stearin) were used: fully-refined paraffin containing 0.5% mineral oil (defined by standard method ASTM D 721) and semi-refined paraffin containing 1%. The paraffin predominantly contains linear hydrocarbons with a carbon chain distribution of C18–C40 (based on GC analysis on an Agilent 7820A system, Santa-Clara, CA, USA). The stearin is composed of free fatty acids, i.e. 50% palmitic acid and 50% stearic acid (based on GC analysis on an Agilent 7820A system, Santa-Clara, CA, USA). Five mixtures (M1-M5) formulated by the experimental second-order central composite design realized by Statgraphics Centurion 17.1.06 (Graphics Software System, STCC, Inc. USA.) were used in our study, as illustrated in Fig. 1.

The composition and characteristics of the blends are given in Table 1. Melting point of the mixtures varied between 48 and 54 °C

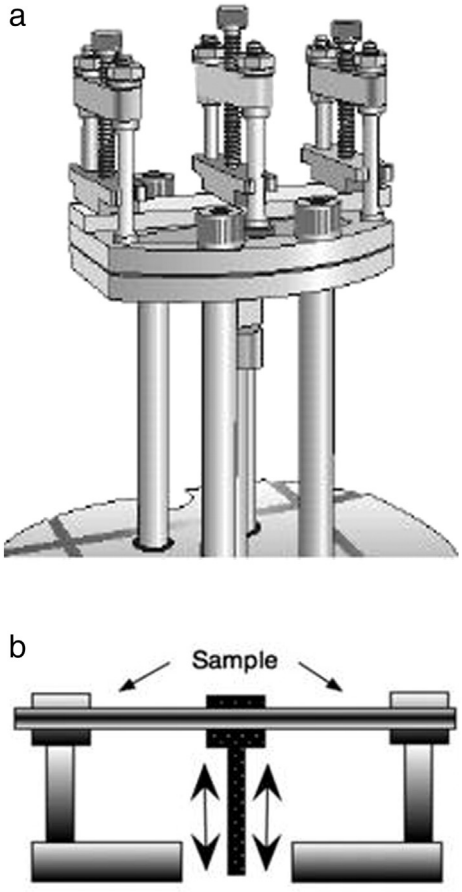

Fig. 2. a) 3-point bending dual cantilever device (TA Instruments Q800 DMA); b) Scheme of dual cantilever device used for rupture test.