

# Gas assisted mechanical expression of cocoa butter from cocoa nibs and edible oils from oilseeds

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## Abstract

The current methods used to recover high quality oil from oilseeds have low yields (mechanical expression, aqueous extraction), require the use of toxic chemicals and rigorous purification processes that can reduce the quality of the oil (solvent extraction with hexane) or are unsuitable for the recovery of commodity oils due to the low solubility of plant oils in environmentally benign solvents (supercritical extraction with CO<sub>2</sub>). Gas assisted mechanical expression (GAME) utilises the much higher solubility of supercritical CO<sub>2</sub> in the oil to enhance the extraction yields of mechanical expression. GAME experiments with cocoa nibs were performed at 40–100 °C, CO<sub>2</sub> pressures of 0–20 MPa and effective mechanical pressures of 20–50 MPa. The maximum yield with conventional expression (71.8%) was obtained at a mechanical pressure of 50 MPa and a temperature of 100 °C. It is shown that GAME has a substantially higher yield than conventional mechanical expression for the recovery of cocoa butter from cocoa nibs, with the highest yield (87.1%) obtained at 100 °C, a CO<sub>2</sub> pressure of 10 MPa and an effective mechanical pressure of 50 MPa. The cocoa butter yield increases with increasing CO<sub>2</sub> pressure until 10 MPa but remains almost constant for higher CO<sub>2</sub> pressures. In contrast to conventional expression GAME also allows the recovery of cocoa butter from cocoa nibs at temperatures below the melting point of pure cocoa butter. The cocoa butter produced with GAME was found to be unfractionated and is therefore of the same quality as mechanically expressed cocoa butter. Experiments with linseed and sesame seed show that GAME performed at 40 °C with 10 MPa CO<sub>2</sub> also results in an increased yield of oil (71.8–83.8% for linseed, 74.3–80.2% for sesame seed) when compared to the yield obtained with conventional mechanical expression performed at 40 °C (38.5–45.7% for linseed, 60.1–65.6% for sesame seed). From these results it is concluded that GAME offers a promising process for the recovery of high-quality vegetable oils at high yields.

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## 1. Introduction

Vegetable oils are valuable raw materials that are widely used in the food, pharmaceutical and cosmetic industries. Currently good quality oil is recovered from oilseeds by performing mechanical expression (separation of liquid from a solid–liquid mixture by mechanical compression) in hydraulic filter presses or screw presses [1–5]. Although this process delivers high-quality, unadulterated oils, the economically obtainable yield (defined as the mass of oil recovered as a percentage of the total mass of oil in the oilseeds) is low compared to that of solvent extraction [1,2,5,6]. Aqueous extraction is used in many rural oil extraction processes. Aqueous extraction yields oil of high quality that does not need degumming, but the yield obtain-

able with this process is relatively low [7]. The use of certain enzymes to weaken the cell walls and thereby freeing the oil can increase the yield, but the cost of the enzymes, as well as the hygiene requirements of a wet process and the cost of demulsification and water removal from the final products have a negative impact on the industrial feasibility of enzyme assisted aqueous extraction [7–10]. Solvent extraction is normally performed with hexane, a toxic and flammable organic solvent [11]. Solvent extraction can however yield oils with different compositions due to the co-extraction of non-fat components. In addition solvent extraction requires rigorous, energy-intensive solvent recovery processes to lower the solvent levels to acceptable levels in both the oil and the solid residue. These processes remove desirable components (e.g. some anti-oxidants and volatile substances responsible for certain taste and flavour characteristics), thereby changing the quality of the oil [5,12]. Alternatively, extraction with environmentally benign solvents such as supercritical carbon dioxide (SC-CO<sub>2</sub>) can be used to produce oil

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Table 1  
Positioning of oil recovery processes with regards to cocoa in terms of product quality, oil yield and commercial use

Process	Oil quality	Solids residue quality	Oil yield	Commercial use
Mechanical expression	++	++	+	++
Aqueous extraction	+	+	–	–
Solvent extraction	–	–	++	+
SC-CO <sub>2</sub> extraction	++	++	++	–
Ideal process	++	++	++	+ to ++

(+) Indicates good, (++) excellent and (–) indicates poor to very poor.

at high yields [11,13,14]. The oil produced in this way is of high quality, but can be fractionated when the process conditions favour selective extraction of certain triglyceride fractions [13,15–24]. Supercritical extraction utilises the solubility of the vegetable oil in SC-CO<sub>2</sub>. The solubility is dependent on the oil under consideration as well as the temperature and pressure, but generally it is in the order of only 1–5 wt.% under normal extraction conditions (25–30 MPa, 40–60 °C) [12–14,25]. Supercritical extraction therefore requires the use of large volumes of carbon dioxide [24,26] and is consequently only feasible for high value, low volume speciality products like essential oils and flavours [17,22]. There is a need for a new process that combines the advantages of the current industrial methods without their respective disadvantages. The positioning of such a process in terms of product quality, oil yield and its suitability for commercial use on a large scale is shown in Table 1.

In this work a new process called Gas Assisted Mechanical Expression (GAME) is introduced. GAME has the potential to overcome some of the disadvantages of other technologies as discussed above. In GAME mechanical expression is performed on oilseed material that has been saturated with SC-CO<sub>2</sub>. Therefore a gas-expanded liquid (the CO<sub>2</sub>-saturated oil) is expressed from the oilseed material. The solubility of SC-CO<sub>2</sub> in vegetable oils is considerably higher than the solubility of the oils in SC-CO<sub>2</sub>; up to 50 wt.% depending on the temperature, pressure and type of oil [12,13]. In consequence a much smaller volume of SC-CO<sub>2</sub> is needed than in supercritical extraction. The viscosity of CO<sub>2</sub>-saturated oils is considerably lower than that of the pure oils at the same temperature [26,27]. Therefore less energy will be needed to express CO<sub>2</sub>-saturated oils from oilseeds compared to the expression of pure oils from the oilseeds. The dissolving SC-CO<sub>2</sub> causes the oil to swell, and consequently CO<sub>2</sub>-saturated vegetable oils occupy a larger volume than the pure oils due to the dissolved CO<sub>2</sub>. This causes the cell structures containing the oil to swell when SC-CO<sub>2</sub> dissolves in the oil, eventually causing these structures to rupture due to their low mechanical strength, thereby freeing the oil. During expression only oil liberated from the cell structure can be recovered: the higher the proportion of the oil contained in the seeds that are liberated from the cell structure, the higher the yield will be. The decreased liquid viscosity and high percentage of the oil freed from the cell structure enhance the recovery of oil in GAME. It is expected that a higher SC-CO<sub>2</sub> solubility in the oil will result in a higher oil yield. Solids often melt substantially below their atmospheric melting points in the presence of SC-CO<sub>2</sub> [28]. GAME therefore offers the additional advantage that oilseeds containing lipid components with atmospheric melt-

ing points higher than ambient temperature can be processed at lower temperatures than those currently used in conventional expression.

The main objective of this work was to experimentally compare the performance of GAME with conventional expression for the recovery of cocoa butter from cocoa nibs (broken cocoa beans). Cocoa nibs contain between 50 and 55 wt.% cocoa butter [29,30]. Cocoa butter is a complex mixture of triacylglycerols (TAG), with 1,3-dipalmitoyl-2-oleoylglycerol (POP), 1-palmitoyl-2-oleoyl-3-stearoylglycerol (POS) and 1,3-stearoyl-2-oleoylglycerol (SOS) accounting for more than 90% of the TAG [15,16,31]. The possibility therefore exists that one, or more, of the TAG will be preferentially extracted when SC-CO<sub>2</sub> is used. Due to the presence of several TAG cocoa butter has polymorphic behaviour, but it is generally accepted that it is completely melted at 35 °C at atmospheric conditions [29,30]. This places a lower limit on the temperature that can be used in conventional expression. It is known that the applied mechanical pressure as well as the temperature has an influence on the yield obtainable with conventional expression [1,32–38]. The CO<sub>2</sub> pressure and the process temperature have an influence on the solubility of CO<sub>2</sub> in the cocoa butter [39]. The applied mechanical pressure, temperature and CO<sub>2</sub> pressure are therefore important process parameters, and their influence on both the yield and quality of the processed cocoa butter will be evaluated. The quality of the GAME recovered cocoa butter will be determined by comparing the composition of this butter to that of mechanically expressed cocoa butter from the same batch of nibs. Furthermore the generic feasibility of GAME will be investigated by comparing the yields obtained with GAME to that of conventional expression for the recovery of oils from linseed and de-hulled sesame seed. Finally the experimental results are used to explain the mechanism of GAME.

## 2. Materials and methods

### 2.1. Materials

Winnowed cocoa nibs were obtained from Gerken's Cacao (Wormer, The Netherlands) while linseed and de-hulled sesame seed were donated by Dipasa (Enschede, The Netherlands). The moisture contents were determined according to the DGF standard method [40]. The original moisture contents of the different oilseeds are reported in Table 2. The fat contents of the different oilseeds, as determined by soxhlet extraction with petroleum ether (boiling range 40–60 °C, Merck, Amsterdam, The Netherlands), are also shown in Table 2. All fat contents are reported on

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