

Preparation of a mixture of cafestol and kahweol

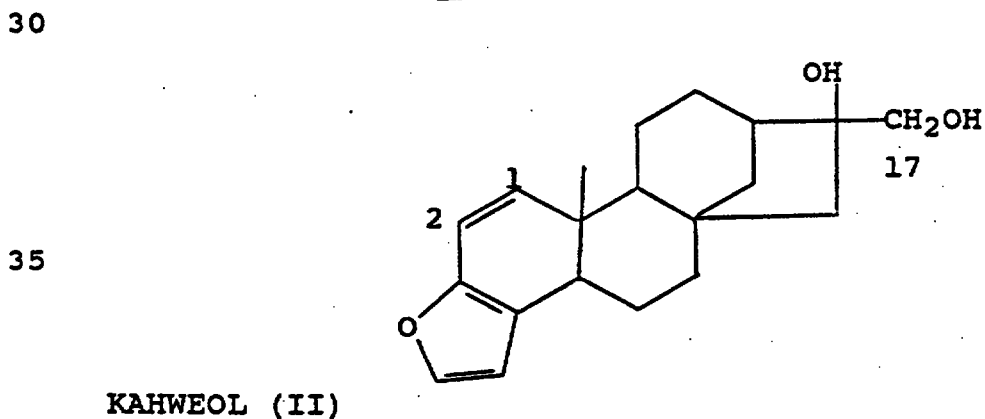
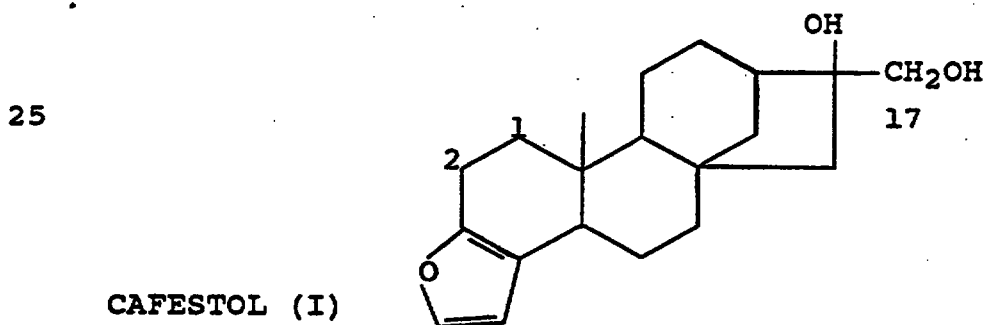
The present invention relates to the preparation of a mixture of cafestol and kahweol derived from coffee oil.

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Coffee oil comprises significant amounts of the esters of two furanic diterpenes : cafestol and kahweol. The actual amounts present vary according to the origin of the coffee : for example, there is no kahweol present in Robusta coffee while Arabica contains a mixture of kahweol and cafestol in the approximate ratio of 1:3. The average composition of coffee oil is as follows:

15	Triglycerides	75-80%
	Esters of cafestol and kahweol	7-15%
	Steroids	2-3 %
	Phosphatides	1-5 %

20 The formulae of cafestol and kahweol are indicated by the following formulae I and II respectively:



It can readily be seen that the only difference between the two compounds is the presence of a double bond between positions 1 and 2 in kahweol. The esters of the cafestol and kahweol present in coffee oil are mainly the palmitates and the lineoleates in position 17.

Crude mixtures of cafestol and kahweol derived from coffee oil may contain between about 15 and 50% kahweol and more usually from 20 to 45% kahweol. These mixtures may be obtained by hydrolysis of the coffee oil followed by the selective extraction of cafestol and kahweol e.g. saponification followed by extraction of the unsaponifiables by a solvent such as methylene dichloride. However, such methods require extremely large volumes of solvent and heating at about 80°C for a prolonged period which can cause the decomposition of the kahweol owing to its heat-sensitivity.

Our copending application No 2188628 provides a process for the preparation of cafestol from kahweol. Preferably the kahweol is present in a crude mixture which comprises cafestol and kahweol derived from coffee oil.

We have developed an improved method of obtaining a mixture of cafestol and kahweol from coffee oil which involves a transesterification process using anhydrous methanol in the presence of a basic catalyst followed by extraction. The use of the methanol enables the process to be carried out at ambient temperature and thus avoiding any thermal decomposition of the kahweol.

Accordingly, the present invention provides a process for preparing a mixture of cafestol and kahweol characterised in that coffee oil is treated with anhydrous methanol in the presence of a basic catalyst, the reaction medium is diluted with a substantially water-insoluble solvent which dissolves the formed

methyl esters and then extracted with aqueous methanol, the aqueous methanol phase then being separated and
5 further extracted with a low-boiling chlorinated hydrocarbon which is then separated from the aqueous methanol phase and evaporated to give the mixture of cafestol and kahweol.

The treatment of the coffee oil with the anhydrous methanol is preferably carried out at ambient temperature with agitation for a minimum of 1 hour, preferably from 2 to 6 hours. The amount of methanol used is conveniently from 20% to 100%, preferably from 30% and 80% and especially from 40% to 60% by weight based on the weight of coffee oil.

10 The basic catalyst is suitably sodium hydroxide, potassium hydroxide or potassium carbonate and the amount used may be from 0.1 to 10%, preferably from 0.5 to 5% and especially from 1% to 4% by weight based on the weight of coffee oil.

15 In the treatment of the coffee oil with anhydrous methanol in the presence of a basic catalyst, the triglycerides and the esters are converted into the fatty acid methyl esters and alcohols (glycerol, sterols, cafestol and kahweol). The substantially water-insoluble solvent which dissolves the fatty acid methyl esters should be liquid under the conditions of the reaction and may be a C₅-C₁₄ alkane, for example, heptane. The amount of solvent is conveniently from 0.5 to 3 parts and preferably from 1 to 2 parts by weight based on the weight of coffee oil.

25 The aqueous methanol preferably contains from about 2% to 15% and preferably from 4% to 12% by weight of water based on the weight of methanol. The aqueous methanol phase contains the cafestol, kahweol, glycerol and the basic catalyst in solution. When the aqueous methanol phase has been separated, it is then extracted with the low-boiling chlorinated hydrocarbon to permit the 30 cafestol and kahweol to be isolated. The low-boiling chlorinated hydrocarbon is preferably dichloromethane.

35 Kahweol possesses an interesting ultra-violet

absorption spectra and the mixture of cafestol and kahweol can be used in cosmetic applications either in the crude form obtained as above or after recrystallisation. Cafestol has anti-inflammatory properties.

The following Examples further illustrate the present invention.

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Example 1

Preparation of a mixture of cafestol and kahweol

15 1000 g of coffee oil containing 11.3% of a mixture of esters of cafestol and kahweol were collected from the spent grounds obtained in the production of instant coffee. To this coffee oil were added 600 ml anhydrous methanol containing 5% by weight of potassium hydroxide
20 and the mixture stirred for 2 hours under nitrogen at ambient temperature. In this stage the triglycerides and the esters of cafestol and kahweol are converted to fattyacid methyl esters and alcohols (glycerol, cafestol, kahweol and sterols).

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After two hours stirring the reaction mixture was diluted with 1500 ml heptane and then extracted with 1540 ml of aqueous methanol containing 10% water with agitation. The heptanoic phase containing the fatty
30 acid methyl esters was then separated from the aqueous methanol phase which has solubilised the cafestol and kahweol, the glycerol and the potassium hydroxide. The extraction was repeated twice using each time 1500 ml aqueous methanol containing 5% water and the three
35 extracts were combined and concentrated under vacuum to 1200 g. 50 g of potassium hydroxide were dissolved in the concentrated extract and agitated at 40°C under nitrogen for 30 minutes after which 2 litres of water

were added. This solution was then extracted five times with a mixture containing 1800 ml of dichloromethane to which had been added 8% by weight of methanol, and the extract evaporated to dryness to give 64 g of a crude orange-coloured mixture of cafestol and kahweol of 70% purity.

This crude mixture of cafestol and kahweol was then dissolved in 500 ml ethyl acetate and the solution agitated for 1 hour with 30 g of active carbon. The solution was then filtered and evaporated to dryness to give 57 g of an orange-yellow oil which was recrystallised in warm methanol to give 50 g of a mixture of cafestol and kahweol containing 40.7% kahweol.

Claims

1. A process for preparing a mixture of cafestol and kahweol characterised in that coffee oil is treated
5 with anhydrous methanol in the presence of a basic catalyst, the reaction medium is diluted with a substantially water-insoluble solvent which dissolves the formed methyl esters and then extracted with aqueous methanol, the aqueous methanol phase then being
10 separated and then further extracted with a low-boiling chlorinated hydrocarbon which is then separated from the aqueous methanol phase and evaporated to give the mixture of cafestol and kahweol.
- 15 2. A process according to claim 1 characterised in that the amount of anhydrous methanol used is from 30% to 80% by weight based on the weight of coffee oil.
- 20 3. A process according to claim 1 or 2 characterised in that the basic catalyst is sodium hydroxide, potassium hydroxide or potassium carbonate.
- 25 4. A process according to any of claims 1 to 3 characterised in that the amount of basic catalyst used is from 0.5% to 5% by weight based on the weight of coffee oil.
- 30 5. A process according to any of claims 1 to 4 characterised in that the substantially water-insoluble solvent is a C₅-C₁₄ alkane.
6. A process according to any of claims 1 to 5 characterised in that the low-boiling chlorinated hydrocarbon solvent is dichloromethane.

