

QUALITY CONTROL OF ZINGIBER OFFICINALE OILS IN PRODUCT FORMULATIONS

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Graphical abstract



Abstract

Zingiber officinale (ginger) is one of the most commonly used flavouring ingredients in food and culinary dishes. The essential oils are known for its unique aroma and characteristic and widely used as therapy. In this study, the essential oil was formulated as massage oil and the quality of massage oil was evaluated on its physicochemical properties, i.e. colour, odour, density, viscosity, refractive index and pH value. The efficacy of the product was determined by the value of the melanin (coloured pigment) and erythema (redness of skin) as well skin irritation analysis (Finn Chamber Patch). The massage oil was also tested using Head Space-Solid Phase Microextraction-Gas Chromatography Mass Spectrometry (HS-SPME-GCMS) to determine the presence of ginger's essential oils important chemical constituents in the massage oil. Analysis revealed the presence of α -zingiberene, neral and geranial which are the main components found in ginger massage oil. For safety use, the finished product was tested for microbial enumeration test and heavy metal analysis. Result showed that there was no microbial contamination and meets the British Pharmacopoeia 2011 specifications in topical use. Meanwhile the results of limit test for heavy metals were found to be below detectable levels of National Pharmaceutical Control Bureau specifications.

Keywords: *Zingiber officinale*, quality control, essential oil

Abstrak

Zingiber officinale (halia) adalah salah satu daripada bahan perisa yang biasa digunakan di dalam makanan dan masakan. Minyak pati halia terkenal dengan aroma yang unik dan ciri-ciri khusus serta digunakan secara meluas sebagai terapi. Dalam kajian ini, minyak pati halia telah dirumus sebagai minyak urut dan kualiti minyak urut dinilai daripada sifat kimiafizik, iaitu warna, bau, ketumpatan, kelikatan, indeks biasan dan nilai pH. Keberkesanan produk telah ditentukan oleh nilai melanin (pigmen berwarna) dan erythema (kemerahan kulit) dan juga analisis kerengsaan kulit (Finn Chamber Patch). Minyak urut halia juga telah diuji menggunakan Head Space-Solid Phase Microextraction-Gas Chromatography Mass Spectrometry (HS-SPME-GCMS) untuk menentukan kehadiran komponen kimia yang penting bagi minyak pati halia di dalam minyak urut. Analisis ini mendedahkan kehadiran α -zingiberena, neral dan geranial yang merupakan komponen kimia utama yang terdapat di dalam minyak urut halia. Untuk tujuan keselamatan, produk siap telah diuji untuk ujian bebanan mikroorganisma dan analisis logam berat. Keputusan menunjukkan bahawa tiada pencemaran mikroorganisma dan memenuhi spesifikasi British Pharmacopoeia 2011 untuk kegunaan luaran. Sementara itu, keputusan ujian had untuk logam berat menunjukkan had logam berat minyak urut halia ini adalah rendah dan menepati spesifikasi Biro Pengawasan Farmaseutikal Kebangsaan.

Kata kunci: *Zingiber officinale*, kawalan kualiti, minyak pati

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1.0 INTRODUCTION

Quality control (QC) is an essential process in product development. QC ensures the safety and efficacy of a product before placing it in the market for consumer usage. By performing QC, the herbal benefits in product will be enhanced. The process of QC in a product development covers every stages, starting with selection of safe raw material, presence of important chemical constituents in final product that contribute to the distinguishable product, consumer acceptance and preference in the product, consistency in batches of production and safety of final product. By assuring the product's quality, consumers become more confident in the product. Thus, high-end product can be penetrating into global market through ensuring the QC.

Natural Product Division, FRIM and Entrepreneur Development Division, MARA have initiated a collaboration namely as 'Program Teknousahawan MARA-FRIM'. The objective of this programme is to assist entrepreneurs in producing a quality product that comply the regulations thus be able to penetrate domestic and international markets. In this study, KUEM Global Sdn. Bhd., one of the participated entrepreneurs had developed *Zingiber officinale* (ginger) massage oil using FRIM technology and quality of final product was determined.

Ginger is one of 18 herbal species that been recognized and has potential to be developed as high-value herbal product under the National Key Economic Area (NKEA EPP#1). This is due to the volatile oil, fixed oil, pungent compounds, resins, starch, protein and minerals contained in the ginger [1]. The characteristic organoleptic properties are contributed by the volatile oil and nonvolatile solvent-extractable pungent compounds. In Malaysia, ginger has been widely used in dishes and drinks as flavouring agent and some of known traditional medicine practices using ginger are as wind expellant and stomach ache treatment [2, 3].

Essential oils are oily aromatic liquids extracted from aromatic plant materials. According to the European Pharmacopoeia 7th edition, essential oils are defined as odorant product, generally of a complex composition, obtained from a botanically defined plant raw material, either by driving by steam of water, either by dry distillation or by a suitable mechanical method without heating. An essential oil is usually separated from the aqueous phase by a physical method that does not lead to significant change in its chemical composition. Essential oils could be then subjected to an appropriate further treatment. They are commercially called as deterpented, desesquiterpenated, rectified or private from "x" according to 7th edition of the European Pharmacopoeia [4]. Essential oil obtained from ginger was also screened for some of pharmacological potentials such as pain management and fatigue. The essential oil is generally safe with minimum adverse effects. Several of these have been approved as food additives and

fall in the category of generally recognized as safe by the U.S. Food and Drug Administration [5].

2.0 EXPERIMENTAL

2.1 Plant Materials, Essential Oil Distillation and Massage Oil Formulation

Rhizomes of eight months old ginger were obtained from Janda Baik, Pahang. Fresh ginger rhizomes were thoroughly washed and cut into small pieces. Essential oils were extracted by Clevenger-type apparatus through hydro-distillation technique. Obtained essential oils were used in formulating massage oil [6].

2.2 Physical Properties Analysis

In order to determine the quality of ginger massage oil, the characteristic of the final product was defined. These characteristics are been used as specification for the final product, massage oil.

1) Colour and Odour

Colour and odour of the massage oil was recorded based on physical appearance observation.

2) Density, Viscosity and Refractive Index

Density of the product was determined using specific gravity meter (Model da – 130 Kyoto Electronics). Viscosity of the product was evaluated by shear stress test using rheometer (TA instrument). Refractive index of the product was checked by using refractometer (Ar 2008 Kruss, Germany) at 25°C.

3) pH Value

pH value of the product was directly recorded by pH meter (Mettler Toledo). The measurements were repeated three times and the range of the value was recorded.

2.3 Determination of Melanin (Coloured Pigment) and Erythema (Redness of Skin) Values and Skin Irritation Analysis (Finn Chamber Patch)

The efficacy of the massage oil was evaluated based on differences of melanin and erythema values before and after massage oil application. The safety of the massage oil's was evaluated by performing skin irritation analysis using *Finn Chamber Patch*. Eight healthy subjects and those without skin allergies were chosen in performing these tests after informed consent. The methodology was conducted in accordance of insult single test described by Jibry and Murdan (2004). Melanin and erythema values were recorded by Skin Analyzer MPA 580 with mexameter probe (Courage+Kazaka, Germany) [7].

The procedure started with marking skin area on the forearm to be tested. Then, the skin was analysed using the mexameter probe to determine the values of melanin and erythema without treatment. Then, the massage oil was rubbed on the same marked skin area and was left for an hour. After an hour, the same skin area was analysed again to obtain readings of melanin and erythema values. The efficacies were determined by comparing readings before and after the massage oil was applied.

Skin irritation analysis was determined using Finn Chamber Patch test by the same procedure, insult single test. However, instead of rubbing the massage oil on the skin, the massage oil was placed using Finn Chamber Patch and left in contact with skin for one hour. After one hour, the site of massage oil application was carefully wiped with a damp tissue to remove any residual formulation from the skin before any assessments or measurements were made. The colour of the skin was differentiate by using red colour scale; + indicates low irritation, ++ indicates medium irritation and +++ indicates high irritation.

2.4 Chemical Analysis

Chemical analysis of massage oil was carried out by using Head Space-Solid Phase Microextraction-Gas Chromatography Mass Spectrometry (HS-SPME-GCMS) Polydimethylsiloxane (PDMS) fibre by GCMS MSD 7890A/5975C (Agilent). The capillary column was a HP-5ms (30 m x 0.25 mm x 0.25 mm). Carrier gas was helium, at a flow rate of 1mL/min; interface temperature at 250°C. The initial temperature was held at 60°C for 10 minutes, then increased up to 230°C with 3°C/min increments and held at this temperature for 1 minute. The chemical components were identified by (HPCH2205.L; Wiley7Nist05.L; NIST05a.L) mass spectra.

2.5 Heavy Metal Analysis

The concentrations of heavy metals in massage oil were determined by using Perkin Elmer Model Analyst 600 Atomic Absorption Spectrometry (AAS). Graphite Furnace Atomic Absorption Spectrometry (GFAAS) was used to measure lead and cadmium metals, while Flow Injection for Atomic Spectroscopy System (FIAS 100) was used to determine arsenic and mercury metals. This AAS system is equipped with hollow cathode lamp (HCL) for cadmium and electrode less discharge lamp (EDL) for lead, mercury and arsenic as the source of the energy.

Heavy metal concentration in massage oil must comply and did not exceed maximum limit as regulated by National Pharmaceutical Control Bureau (NPCB) in Drug Registration Guidance Document (DRGD), First Edition, January 2013 Revised July 2015 [8]. The maximum limits for heavy metals are as follows (Table 1);

Table 1 The maximum limit for heavy metals in Drug Registration Guidance Document (DRGD), First Edition, January 2013, revised July 2015

Heavy metal elements	Maximum limit (mg/kg or mg/litre or ppm)
Lead	NMT 10.0
Cadmium	NMT 0.3
Mercury	NMT 0.5
Arsenic	NMT 5.0

*NMT= Not more than

2.6 Microbial Contamination Analysis

Total aerobic microbial count (TAMC) and total yeasts and moulds count (TYMC) in the formulated ginger massage oil was determined according British Pharmacopoeia to fulfill the quality specification by DRGD. Acceptance criteria of TAMC and TYMC were 2×10^2 CFU/mL and 2×10^1 CFU/mL, respectively.

3.0 RESULTS AND DISCUSSION

Essential oils from hydrodistillation process are dark yellow in colour and the yield was 0.47%-0.69%. Hydrodistillation process will form solid-liquid mixture when it was heated until boiling under atmospheric pressure in an alembic, where the heat allows the release of odorous molecules in plant cells. These volatile aroma compounds and water form an azeotropic mixture [9]. The essential oil is on the top of the mixture and was separated to be formulated into massage oil due to the refreshing yet calming aroma.

As part of QC of ginger massage oil, the physical properties analysis was conducted. Colour of the formulated ginger oil was light yellow and transparent. Meanwhile the odour was spicy and lemony. Density of massage oil was recorded at 0.9104 - 0.9108. Its viscosity is low due to its aspiration in producing massage oil that is not too sticky thus does not leave an uncomfortable feels on the skin [7]. Viscosity was measured by the resistance of a fluid which is being deformed by either stress or tensional stress [7]. The thin oil is ideal for the aromatherapy and cosmetic industries as it has a light texture and is easily absorbed into the skin [10].

The refractive index of massage oil at 25°C was recorded in the range of 1.440 - 1.4600. The importance of identifying the refractive index of the products is to determine the translucency of the oil. Refractive index (n) of a medium is defined as the ratio of the velocity, c of a wave phenomenon such as light or sound in a reference medium to the phase velocity, V_p in the medium itself [11]. pH Value of the formulated ginger oil is in the range of 5.12 – 6.50. This values falls into the safe range of pH value of skin which is 5.0 - 6.5.

Almost 62.5% of the eight subjects showed a decrease in melanin values, in response of the formulated ginger oil has the potential to brighten

the skin. Meanwhile, 75% showed an increased of erythema values due to the potentials to redden the skin. However the redness may occur due to increased of skin surface's blood flow. The skin irritation test also supported that there is no irritation effects were detected in any subjects.

The chemical composition of the formulated ginger oil by HS-SPME-GCMS analysis revealed the presence of eugenol (50.94%), *eugenol acetate* (7.08%), geranial (4.90%), *α -zingiberene* (4.83%), *β -caryophyllene* (4.68%) and neral (4.04%).

Quantification of heavy metals analysis (Table 2) revealed that the amount of lead presence still below the regulated limit. Meanwhile cadmium, arsenic and mercury showed to be less than 0.01 mg/kg.

Table 2 Heavy metal contents in formulated ginger oil

No	Parameter	Results (mg/kg)
1	Lead (Pb)	0.03
2	Cadmium (Cd)	ND (<0.01)
3	Mercury (Hg)	ND (<0.01)
4	Arsenic (As)	ND (<0.01)

* ND = Not detected; Less than the minimum detection limit reported

Total aerobic microbial count (TAMC) and total yeasts and moulds count (TYMC) growth only less than 10 colony forming units/mL. This result appears to be within the limits of permissible levels stipulated by NPCB.

4.0 CONCLUSION

The formulated ginger massage oil has been successfully registered with the National Pharmaceutical Control Bureau (NPCB) and gets the notification number, thus it allowed to be sold into the market. The product has meets the specifications regulated by NPCB is in accordance with the requirements set forth in the Poisons Act 1952 and its Regulations, Sales of Drugs Act 1952 and the Control of Drugs and Cosmetics Regulations 1984. Based on the studies that have been conducted, this product is proven effective and safe to be used through positive results from several tests including its physicochemical properties, value of the melanin (coloured pigment) and erythema (redness of skin)

as well skin irritation analysis (Finn Chamber). In addition, the results of heavy metal test and microbial load analysis were below the regulated limit by NPCB.

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