Microwave assisted hydro-distillation of essential oils from fresh ginger root (*Zingiber officinale* Roscoe)

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ABSTRACT

A solvent free in situ microwave hydro-distillation method for extraction of essential oil from fresh ginger root it presented. Extraction was conducted in a TE_{10n} single-mode microwave cavity and variable power 2 kW generator operating at 2.45GHz. The main extracted components identified by gas chromatography (GC) were Zingiberene, α-Curcumene, β-Sesquiphellandrene and α-Selinene. At energy inputs of 0.40 kWh/kg higher powers and shorter exposure times, crucially did not degrade the highly volatile components (α-Pinene and Camphene) despite providing the highest essential oil yields. Optimum processing conditions were found to be 1000W (0.40kWh/kg) for 5 min, for whole ginger root, where 0.35g oil/100g plant was obtained. This was compared to a yield of 0.2g/100g plant in 150 min in using conventional hydro-distillation and 0.3g/100g plant in 90 min using a multi-mode microwave cavity-based hydro-distillation.

Keywords: *ginger essential oil, microwave assisted extraction, solvent free, GC.*

1. Introduction

Essential oils, also known as essences or volatile oils, are substances biosynthesized by living organisms. They can be liberated from the material which contains them by distillation, pressing or extraction with a suitable solvent. They have found uses in fragrance, food, cosmetic and pharmaceutical industry [1, 2]. The composition of the essential oils varies according to the region they are cultivated in and to the climate conditions [3-7]. Also, the various methods for drying ginger before oil extraction affect the ginger oil composition [8, 9]. Besides their pleasant odors, essential oils are valuable products due to the bioactive properties they possess [10-14].

Essential oils can be obtained through different conventional methods such as: steam distillation; hydro-diffusion; hydro-distillation; destructive distillation; cold expression; as well as novel innovative techniques such as supercritical fluid extraction [15]; turbo distillation; ultrasound-assisted extraction; microwave-

assisted extraction; and instantaneous controlled pressure drop technology [2, 16]. However, the literature reports that the conventional methods can affect the final product quality, due to losses of some volatile compounds during the procedure, low extraction efficiency, and degradation of unsaturated compounds due to thermal effects or solvent used in the extraction [17, 18].

Considering the extraction of essential oils, microwave-assisted processes are highly desirable due to their small equipment size, simplicity and rapid controllability through mild increments of heating. The main benefits of microwave assisted extraction are the decrease of extraction time and solvents used. The advantages of using microwave energy, which is a non-contact heat source, for the extraction of essential oils from plant materials include: more efficient heating; faster energy transfer; reduced thermal gradients; selective heating; reduced equipment size; faster response to process heating control; faster start-up; increased production; and elimination of process steps [19,20].

However, previous work has often focused on the use organic solvents to extract the essential oil contained in dried ginger [21]. Not only would the preparation of the ginger incur additional processing costs in a commercial system, but the solvents used can be costly and harmful to the environment. Other workers have trailed the use of a range of microwave absorbing powders (so called suseptors) mixed into dried ginger powder [22]. But by the addition of a suseptor to the dried plant, it is heated indirectly through thermal conduction, thereby negating some of the principal benefits (those being selective and/or volumetric heating) of microwave processes. Furthermore these studies have been conducted with adapted domestic microwave ovens. These multimode cavities suffer from a number of disadvantages including spatial inhomogeneity of the electric field distribution and the use of relatively low applied powers. Furthermore, without the use of specialist equipment, it is not possible to determine the actual energy absorbed by the sample under test.

In this work we want to evaluate the use of Solvent Free "*in situ*" Microwave Generated Hydro-distillation [23] and Microwave Assisted Hydro-Distillation (MAHD) as a means of extracting the essential oils from ginger root using a bespoke single-mode microwave cavity operating at powers of up to 2kW at a frequency of 2.45GHz [24]. This will be compared to both a domestic multimode oven and conventional hydro-distillation. The effect on the total yield and composition of the essential oil recovered is to be investigated in terms of the experimental parameters used in the microwave based extraction system. These being: applied microwave power; extraction time; total energy input; and starting material preparation.

For determining the optimum microwave treatment conditions in terms of product quality and yield, the dielectric properties of the vegetal material (fresh shredded ginger root, ginger pulp and ginger juice) were determined and correlated with the dimensions of the microwave applicator. This is equipped with mechanical stirring and air bubbling through the plant material for a uniform heating entrainment of the volatile components. The composition of the extracted oil was subsequently analyzed by Gas Chromatography Mass Spectrometry (GC-MS).

In this study, the effect of the MW cavity type (multimode or single mode) was evaluated in relation to the extraction efficiency of the ginger essential oil. Multimode cavities, similar to domestic MW ovens, generate a spatial-inhomogeneity in electric field strength within the volume of the cavity – that is, in certain areas, the electric field strength is higher than in other areas. This makes it difficult to obtain an even treatment of the plant material to the MW energy. Moreover, in this set up, the absorbed and reflected powers cannot be controlled, so the MW energy introduced in the system is not fully used. Often in domestic

MW ovens, for a given applied power, it is cycled on and off automatically during operation, and this cannot be controlled by the user. Single mode cavities present a great advantage because they support spatially well-defined areas of high electric field strength. By using ancillary equipment such as a three-stub automatic tuner for impedance matching and a sliding short circuit, the power absorbed by the sample can be optimized, such that the reflected power is minimal, in addition to facilitating in-line measurement of the scattering parameters (from which absorbed power can be derived). These advantages presented by the single mode cavity allow a very efficient use of MW power. The extraction of the essential oil from the plant material is enhanced resulting in higher yields for a given energy dose. By using this set up, the MW power used can be increased to values in the range of kW, and as the power increases, the extraction time decreases accordingly. However, the power used for extraction must take into consideration the sensible character of essential oil components.

In the case of single mode cavities operating at 2.45 GHz, one of the main drawbacks is the fact that it cannot process large volumes of plant material, as it is limited by small diameter of the reactor. A potential solution to this problem is to develop the extraction process from a batch to a continuous flow process, where the throughput can be increased, and thereby yield of product. In addition, the concept of frequency scaling could also be used to develop the technology as a commercial-scale system. That is, by using electromagnetic energy at a lower frequency, the size of the applicator can be increased, as the dimensions of a single-mode applicator are defined by the first primary mode (wavelength) of propagating energy. Larger single-mode applicators, therefore also offer potentially variable route to scale-up for this technology.

The in-situ MWHD of ginger essential oil from fresh ginger rhizome using an energy efficient, single mode MW cavity not only offers potentially increased yields, with much reduced reaction times, but also offers additional advantages of established extraction techniques. As water already contained within the plant material is used in the extraction, the use of organic solvents, which are harmful to the environment, is not required. This also removes processing steps associated with the management of this waste stream. Furthermore, it maintains the residual solid matter in a form which could be used as a food supplement rich in fiber, an animal feed or as biomass for power generation for example.

2. Materials and methods

2.1. Materials

Fresh ginger root (*Zingiber Officinale* Roscoe) acquired from the market in Nottingham, UK. The plant was shredded using a kitchen blender prior to extraction. A master batch was prepared by mixing together 5 kg of shredded ginger root (2 – 6 mm pellets), so as to provide a uniform sample. This was stored in a refrigerator to minimize sample deterioration prior to use. The effect of a number of feedstock preparation methods on the oil yield was investigated. These are described in section 3.5.

2.2. Methods

The physico-chemical quality of the ginger essential oil obtained (from conventional and MW extraction) was studied and compared to that found in literature.

The moisture content of the ginger subject to the different methods of feed preparation was determined by the ASTA/AOAC distillation method [25, 26].

2.2.1. Conventional Hydro-distillation

Conventional hydro-distillation (CHD) undertaken using a Neo-Clevenger type extraction system. A fresh ginger mass of 200g was used and the essential oil obtained was collected, separated from the water mass by centrifuge and then weighed with an extraction time of 150 minutes.

2.2.2. MAHD – Multimode cavity

The ginger essential oil was obtained through a microwave assisted hydrodistillation process, using an optimized procedure similar to the literature [27]. The plant material used was fresh shredded ginger root. The weight ratio of plant material to distilled water was one to two. The extraction time was of 90 minutes.

2.2.3. SF in situ MHD and MAHD – single mode cavity

The microwave system consists of a TE_{10n} single mode cavity in WR340 waveguide terminating in a short circuit piston tuner used in order to superimpose the incident and reflected waves. This was connected to a variable power Sairem 2kW generator and magnetron head generating microwave energy at 2.45 GHz. An automatic three-stub tuner was used to match the impedance of the transmission line to that of the cavity to ensure reflected power was minimized.

The reactor in which the plant material was placed was made out of Pyrex glass. It comprised of a cylinder with a 70 mm diameter and a height of 150 mm. It was made out of two pieces to enable the addition of the plant material. This reactor was narrowed at both ends and continued in two 10 mm diameter tubes; one at the top and one at the bottom as shown in Figure 1.

Figure 1: Schematic description of the set-up for microwave assisted extraction of essential oil from ginger root.

The 70 mm diameter tube had a frit mounted inside it that was used for holding the plant material in place. The frit was necessary to enable the passing of air through the plant material from the bottom tube of the reactor and out the top tube, thereby entraining the vapors released from the plant material towards condenser. To ensure optimal treatment, the reactor was filled with the plant material to a maximum depth of 55 mm, as this is the internal height of the waveguide used in the microwave system. The extraction was conducted with and without an overhead stirrer, with paddles. The stirrer was necessary in order to ensure a uniform exposure of the plant material to the microwave action and this way to improve the yield in essential oil. The stirrer was introduced through the top of the reactor with the stirrer rod made out of glass and the paddle out of PTFE, both MW transparent materials.

The water vapors entrain the components of the volatile oil and the air flow passed through the reactor must be chosen so as to ease the transport of the vapors towards the condensers and at the same time allow their condensation. Experimentally, the maximum possible value for the air flow, without entrainment of the

plant material from the reactor, was determined to be 10L/minute and was used throughout the experimental programme.

The essential oils were collected using a condenser followed by a series of three condensation traps submerged in ice water (Fig. 1). After the extraction, each trap is weighed separately in addition to the reactor containing the rest of the plant material, so as to measure the efficiency of the experiment. The three condensation traps are washed with 25 ml of hexane, to collect all the oil extracted in the hexane phase, following GC analysis.

The water content of the ginger sample left in the reactor after each extraction was determined by the ASTA/AOAC distillation method.

2.3. Material Characterization

Dielectric property measurements were performed at room temperature using the dielectric coaxial probe (for fresh ground ginger, pressed ginger pulp, ginger juice) and the cavity perturbation technique using a resonant circular copper cavity of diameter 550 mm and height 55mm resonating in TM_{0n0} modes [28,29] (for oven dried ginger).

GC-FID analysis: The composition of the recovered essential oils was analyzed by Gas Chromatography (GC). The GC analyses were performed using a Hewlett Packard chromatograph HP 6890 series GC system; Column type: DB-1 (J&W Scientific) 50 m x 0.25 mm; 0.50 micron; 60 to 325/350°C; SN: U55186943H, FID detector; (Dimethylpolysiloxane); Temperature profile: 40 – 250°C, 2°C/min; Injected volume: 1 µL; Injection/detection temperature: 250°C (Sample: 0.5 mL 0.1% nonane (internal standard) in hexane solution + 0.5 mL essential oil in hexane sample; Split ratio: 10; Range: 0; Area reject: 1).

GC-MS analysis: The GC-MS equipment used for the qualitative analysis of the essential oil was Agilent 5975, composed of a gas chromatographer and a mass spectrometer with a quadruple filter. A DB-EUPAH column was used, with a length of 60 m, an internal diameter of 0.25 mm and a film thickness of 0.25 microns. The carrier gas was helium, with a 1.8 mL/min flow. The temperature program used was as follows: the start temperature was 30°C (3 min) followed by an increase of 4°C/minute until reaching the final temperature of 280°C (10 min). The temperature of the injector was 250°C, that of the transfer line 280°C, and that of the quadrupole was 150°C. The detection of the components was performed in the range of 10 – 500 m/z. Each chromatographic signal was analyzed using MSD Chem Station software and Nist databases for the compound identification. For each analysis, 30 µL of sample were injected.

3. Results and Discussion

The physico-chemical characteristics of the ginger essential oil obtained are presented in Table 1, and they correspond to the specifications presented for ginger essential oil [30].

Table 1: Physico-chemical characteristics of ginger essential oil, obtained by MW and conventional treatment.

Ginger root was studied under different methods of preparation. Experiments were carried out using fresh shredded whole ginger root, using ginger pulp – ginger root pressed of 80% (w/w) of its juice and using

ginger juice, in order to determine the form that offers the best results in terms of both essential oil composition and overall oil yield.

3.1. Moisture content

All the types of ginger used for the experiments were measured for moisture content. It was found that fresh ginger root had a moisture content of 92% (w/w), pressed ginger pulp has a moisture content of 80% (w/w) and the ginger juice has 94.5% (w/w) moisture.

3.2. Dielectric properties for ginger root

The results are presented in Figure 2. The data for water was added so as to enable a comparison. The penetration depth was calculated from the measured dielectric properties according to Equation 1:

$$Dp = \frac{\lambda_0'}{2\pi(2\varepsilon')^{\frac{1}{2}}}\left[(1+\left(\frac{\varepsilon_{eff}^{"}}{\varepsilon'}\right)^{2})^{1/2}-1\right]^{-1/2} \quad \text{(Eq.1)}$$

Where: Dp is the penetration depth (m), λ_0' is the free space wavelength (m), ε' is the relative permittivity, and ε_{eff}" is the effective loss factor [31].

Figure 2: The dielectric properties values measured for whole ginger root, for the two constituents: ginger pulp and juice, for oven dried ginger and water, for comparison, at 2.45 GHz frequency.

As seen from the Figure 2, the lower the water content of the plant material, the greater the penetration depth, indicating that water is the dominant absorbent phase in the material. For oven dried ginger, the penetration depth is 100 times greater than for fresh ground ginger root. However, by drying ginger, some of the highly volatile components essential oil components in the plant are lost, due to the thermal treatment. For fresh ground ginger and ginger pulp, due to the low penetration depth, it is mandatory to ensure stirring of the plant material, to provide an even exposure to the microwave energy.

3.3. Microwave assisted hydro-distillation of ginger essential oil

Initial experiments were conducted on ginger pressed of its juice without stirring in the reactor. The oil yield as a function of applied microwave power and processing time is presented in Table .

Table 2: Essential oil yield obtained from pressed ginger pulp without stirring

These combinations of power and treatment time gave total energy inputs of between 0.14 to 0.36 kWh/kg of pressed ginger material. It can be seen that the maximum yield of 0.22 %was achieved at a power of 450W applied for 5 minutes. This corresponds to a total delivered energy of 036kWh/kg. Experimentally, it was observed that the ginger is thermally degraded at an energy level greater than 0.36 kWh/kg (exp. performed at 450 W), and so therefore sets an upper limit on the total amount of energy that can be delivered during these experiments and, consequently, to the essential oil extracted. As a result, all subsequent experiments were carried out considering this limitation.

It is suggested that given that the calculated penetration depth for the feed material is only 1.27 cm at 2.45 GHz (Figure 2), the total oil yield is greatly dependent on the distribution (and packing density) of the plant material on the incident face of the reactor. Therefore, all further experiments were conducted with stirring of the material in the reactor, in order to homogenize the uniformity of microwave treatment.

3.4. Residual content of essential oil after microwave-assisted extraction

The treated ginger (pulp and whole ginger) was submitted to a further conventional hydrodistillation in order to determine if any residual oil was contained within the plant material. The samples that were submitted to this further testing were one for each MW power (250, 500, 1000 and 2000W) for ginger pulp and whole ginger. No oil was extracted, which confirms that the process used led to an almost complete extraction of the oil.

3.5. The Effect of Feed Preparation on the Yield of Essential Oil

In order to increase the yield of essential oil, the effect of the initial preparation of the feedstock was investigated. The whole ginger root was prepared to the following methods:

1. Shredded un-pressed ginger with the addition of 50ml water – to be able to compare with the results from conventional and multimode extraction
2. Pressed ginger comprising 20% of the mass of the whole root
3. Pressed juice comprising 80% of the mass of the whole root

The ginger was pressed of its juice to reduce the water content of the test sample prior to treatment. 100g of the shredded ginger pulp yielded 80g of juice, leaving a residual 20g of pressed root. As presented in Figure 2 the penetration depth of the microwave energy has an inverse relationship to the water content of the plant material. It then follows that by removing a proportion of the water, more of the plant mass will interact with the applied microwave energy, thereby increasing the efficiency of the SF *in situ* MHD process. For these three preparation methods, the material was subjected to SF *in situ* MHD and MAHD (whole ginger) at increasing applied microwave power between 250 and 2000W, for correspondingly shorter treatment times, such that the total applied energy dose remained constant at ca. 0.40kWh/kg. The plant material was mechanically stirred at 200rpm during the treatment process for all the preparation methods. Stirring rates higher than this cause an entrainment of plant material from the reactor. The results are presented in Figure 3.

Figure 3: Effect of microwave power on the ginger oil yield for whole shredded ginger, and separated ginger juice and pulp at a fixed energy dose of ~0.40 kWh/kg.

Considering the fresh ginger with the addition of an extra 50ml of water, increasing the power only correspondingly increases the yield of essential oil up to some critical point, somewhere between 500 and 1000W for a fixed energy input of 0.40kWh/kg. Applying powers greater than 1000W does not give further increases in oil yield in this case. However, up to this power threshold, it is suggested that the efficiency of the hydro-distillation process of the volatile oils is increased, because the steam distillation/ stripping of the oils in the aqueous phase is more energetic. Heat transfer to the bulk material is also minimized, which has

been shown in other similar microwave based oil extraction systems to be a critical factor for the total yield of oil recovered [32].

Increasing the applied power does correspond to an associated drop in final moisture content (and thereby more water removed from the material). At an applied power of 250W (0.40kWh/kg), the final moisture content of the plant materials was 83%, while at 2000W (0.40kWh/kg) it was 68%. This could suggest that the maximum theoretical yield of oil is in the region of 0.35% (w/w) for this particular preparation of the feedstock material. As the concentration of oil in the ginger decreases, so does the amount removed during the hydro-distillation. Once all the available oil is removed, then only water is distilled from the reactor. This is also supported by the fact that conventional hydro-distillation of the plant residue post-treatment did not yield any further recovery essential oil.

It can be seen from Figure 3that by treating the pressed ginger pulp and associated extracted juice separately, the oil yield is increased from 0.35 to 0.42g oil/ 100g ginger, compared to treating the whole ginger alone. It is suggested the increase in yield is the result of liberating the essential oil from the plant matrix by pressing it, thereby making a greater proportion of the oil available for subsequent MAE extraction. The increased yield may also be attributed to the reduction in water content of the pressed material increasing the microwave penetration depth into the sample. This results in a greater proportion of the material being exposed to microwaves again increasing the treatment efficiency. This increase in the applied power from 1000 to 2000W (0.40kWh/kg) also results in an increase in oil yield from the separated fractions, not observed in the treated whole root. In the pressed material, this could be attributed a more efficient hydro-distillation of the oil fraction resulting from a more energetic process at higher powers.

The essential oil yields obtained from the single mode based system in comparison to those from the multimode cavity experiments and steam distillation are presented in Table 3

Table 3: Comparison of the essential oil yields obtained from the different extraction processes evaluated

Note: * - combined yield processing the pressed plant and juice separately for 2000W applied power at 0.40kWh/kg each.

It can be seen from **Error! Reference source not found.** that using the microwave single mode system results in an increase in the essential oil yield of over 250% compared to conventional steam distillation. A reduction in processing time from 150 minutes (steam distillation) to just 5 minutes (single mode cavity) is also achieved. This also limits the possibility of thermal degradation of the more volatile essential oil components.

3.6. Qualitative and quantitative analysis of ginger oil volatiles

The composition of the essential oil as determined by GC, for the whole ginger, juice and pulp are presented in Figure 4.

Figure 4: Major compounds identified by CG-FID in ginger essential oil obtained from whole ginger root (A), ginger juice (B) and ginger pulp (C) at different powers and at the same energy input: 0.40kWh/kg.

Zingiberene, α-Curcumene, β-Sesquiphellandrene and α-Selinene are the main components identified in the essential oil extracted using the single mode MW extraction cavity. It can be observed that the composition is mostly the same for all power inputs, and this means that high operating powers for short extraction times do not affect the composition of the essential oil (do not degrade it). For higher powers and shorter times, the highly volatile α-Pinene and Camphene are extracted without them being degraded.

Comparing the composition of the oil extracted from ginger juice and pulp, the components are fairly evenly distributed among these two parts of the ginger plant (Figure [4.B, 4.C]). Comparing the results of the microwave extraction to those obtained in conventional extraction (Figure [4.A], Table 4), we can see that the composition is fairly the same, none of the highly volatile compounds are lost and even at high power inputs, the valuable oil is not degraded.

A difference can only be seen for the extraction of the whole ginger root at 250 W (0.40kWh/kg), as it is a low power and the extraction is slower, so the main components extracted are the more volatile ones, this explaining the decrease in Zingiberene and increase in α-Curcumene.

Table 4: Comparison of the essential oil components obtained by GC-MS analysis of ginger oil obtained by SF in situ MHD and by CHD.

3.7. Energy requirements for the extraction of essential oil

It was shown in section 3.5 that the yield of essential oil can be increased if the plant material is mechanically pressed into separated fractions comprising of its pulp and extracted juice. However, this represents an additional processing step and also two separate microwave treatments. In order to determine if treating the arising fractions separately is advantageous, an evaluation of the energy balance was undertaken and is presented in the following section.

The amount of liquid (oil + water) distilled during the microwave treatment is given by Eq. 2:

$$M_w = M_i - M_f \qquad \text{(Eq.2)}$$

Where: M_w is mass water removed (g); M_i is the initial plant mass (g); and M_f is the final plant mass after extraction (g)

The energy required to remove a given mass of water is then calculated according to Eq. 3:

$$Q = h_v M_w \qquad \text{(Eq.3)}$$

Where: Q is energy (kJ); h_v is latent heat of vaporization for water (2260J/g); and M_w is mass water removed (g)

It has been shown previously that the yield of extracted oil is proportional the amount of water which is vaporized during the hydrodistillation process. The essential oil yield recovered from processed material subjected to the three methods of preparation is shown in Figure 5, as a function of the energy required to

remove the corresponding amount of water in each test. It can be seen from the figure that by processing the pulp and juice separately, the combined yield is highest when the maximum power is used – 2000W (0.40kWh/kg) for the shortest time (total time 3 ½ min). While the yield from the pressed material is low, it should be noted that this only represents a mass of 20g of material, as 100g whole ginger is separated into 20g pressed pulp and 80g recovered juice. Should the whole root be pressed prior to use, then while some oil will be lost to the juice, the remainder is effectively concentrated into the pressed pulp. This coupled with the increased penetration depth resulting in the removal of a large volume of the liquid, results in a more effective treatment method. However, an economic analysis would have to be undertaken to evaluate if the increased yield of essential oil outweighs the costs associated with this extra processing step.

Figure 5: the effect of feedstock preparation method and energy required to vaporise the removed mass of water as a function of essential oil yield.

4. Conclusions

The lower the water contents from the plant material, the better the MW penetration depth. For oven dried ginger, the penetration depth is 40 times better than for fresh ground ginger root. Stirring ensures even exposure of the plant material to the MW action and increases the yield. A maximum energy input must be determined and not exceeded in order to avoid degradation of the plant material which was determined in the present work as 0.40kWh/kg.

Ginger pulp gave the highest yield in essential oil, but represented only 20% of the whole ginger. Conventional extraction offers a yield of 0.2 g oil/100 g plant (150 min), multimode cavity gives 0.3 g oil/100 g plant (90 min) and single mode cavity gives 0.35 g oil/100 g plant (1000W, 0.40kWh/kg, 2.5 minutes).

The main components detected by GC in the essential oil are: Zingiberene, α-Curcumene, β-Sesquiphellandrene and α-Selinene. The composition is mostly the same for all power inputs – high operating powers for short extraction times do not affect the composition of the essential oil (do not degrade it). For higher powers and shorter times, the highly volatile α-Pinene and Camphene are extracted without them being degraded. Comparing the composition of the oil extracted from ginger juice and pulp, the components are fairly evenly distributed among these two parts of the ginger plant.

Overall, for an equivalent energy input, the overall yield of essential oil is proportional to the applied power. This can be attributed to more efficient distillation of the oil fraction when the aqueous phase is heated. Rapid heating increases the volatility of the oil fractions and minimizes heat transfer to the bulk plant material, but crucially, not degrading some of the more volatile components of the essential oil.

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