Production of Plant Extracts by Supercritical Fluid Extraction

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Abstract: A case is presented for the introduction of a flexible commercial Supercritical Fluid Extraction (SFE) operation in Trinidad and Tobago (T&T), whereby waste carbon dioxide from the ammonia industry is used to extract high value products from indigenous plant materials. Typical raw materials would be waste products from food processing such as orange peel and pumpkin seeds, as well as crops which are, or could be, grown locally such as turmeric, hot peppers, ylang ylang and vetiver. New crops could utilise land formerly used for sugar cane or could be supplied from contract farmers. Background research on the extraction characteristics of most of the above potential raw materials has been carried out on bench scale SFE units at The University of the West Indies (UWI). Typical data for pumpkin seeds and ylang ylang is presented, giving extraction curves (% extraction with time), variation of total quantum of extraction with varying processing conditions (operating pressure and temperature), as well as product quality as evidenced by chromatographic analysis. The potential for producing both essential oil and oleoresin separately from turmeric in a single operation, by changing operating conditions during a run, is also presented. A flowsheet for the envisaged process is presented, with an order of magnitude estimate of the capital cost of an operation of a ‘3-extractor, 2-separator’ system being determined to be approximately US$7.5 million. By way of example, the economic feasibility of a plant to produce the extract of turmeric, being marketed at the current world price of US$50 per kg, on the basis of a 310 day per year operation with a 3-hour turn-around time gave an internal rate of return (IRR) of 21% on a 5-year projection. This demonstrated that such an operation could be commercially feasible and a Business Plan should be developed to move the concept forward.

Keywords: Essential oil, oleoresin, carbon dioxide, industry, feasibility, business plan

1. Introduction

Many plant species in the tropics secrete complex chemicals which are extracted and incorporated into various consumer products which may be classified under the following headings:

- Food Flavours
- Aromatherapy/Personal Care products
- Nutraceuticals/Pharmaceuticals
- Perfumes
- Various uses e.g. security sprays, insecticides, dyes and colourings

There are two basic types of plant extracts, essential oils and oleoresins. Essential oils are the volatile oils giving rise to the odour from the plant. Oleoresins, however, have much higher vapour pressure than the essential oils, boiling at higher temperatures. Grand View Research (2014) estimated the global essential oil market to be approximately US$5.5 billion in 2014. They stated that increasing essential oil penetration in aromatherapy, coupled with rising demand for fragrances and flavours in food and beverages, is expected to remain a key driving factor for the global market.

Growing consumer preference for natural products has led to the development of novel applications in personal care and beauty products. Rapid industrialisation and growing disposable incomes, particularly in emerging economies such as China, India, Vietnam and Thailand, are some of the macro factors steering growth. In addition, rising application scope on account of growing consumer awareness regarding health benefits, and negligible side effects associated with the use of essential oils, is expected to spur their demand in the medical industry. Growing demand for aromatic flavours and fragrances in cosmetics, perfumes, as well as spa and relaxation applications is also expected to fuel demand in the coming years.

Specifically, Grand View Research (2016a) estimated that the United States (US) essential oil market revenue will double from ~US$2.0 billion in 2015 to more than US$4.0b in 2022, with breakdowns being given for the top 10 oils. The European market was valued at US$2.4b in 2014, but growth was expected to remain stagnant in that market. The Oleoresin market was estimated by Grand View Research (2016b) to be ~ US$1.14 billion in 2014, increasing to US$1.69 billion in 2022. Shifting consumer preferences towards natural ingredients over synthetic flavours is expected to have a positive impact on the market growth. Oleoresins are also widely used in
treating cancer, stress and back pain owing to their antioxidant and anti-inflammatory properties which is expected to further stimulate its market development.

The Caribbean does have a long history of essential oil production, albeit limited in the range of products. Thus, bay oil has been produced in Dominica since the 19th century and lime oil was produced in a number of the islands for many years (Guenther, 1961). More recently, Pimento Oil production has been introduced in Jamaica, and Nutmeg Oil in Grenada, Anise Oil in Trinidad and Bay Oil in Tobago. Even though these industries are small by international standards, they have contributed significantly to the economy in the small islands of Dominica and Grenada. In the case of Trinidad and Tobago, Anise Oil was produced on old sugar lands at Orange Grove by the Pernod Company for some time before being closed down a few years ago. In addition, a Bay Oil facility was installed in Tobago in the 1970’s to exploit a mature bay tree plantation, but not operated continuously.

Traditionally, essential oils were extracted from the plant materials by steam distillation, whereas oleoresins were extracted by solvent extraction techniques. The more modern method of Supercritical Fluid Extraction (SFE) utilising carbon dioxide as the extraction fluid, however, has the capability of extracting both essential oils and oleoresins, giving rise to a high level of flexibility in operation. The work described in this paper compares SFE with the traditional extraction techniques and demonstrates that it could be used commercially in Trinidad and Tobago in the resuscitation of the industry. The timing of the introduction of the industry in Trinidad and Tobago is opportune, with the need to diversify the economy and the opportunity to exploit expanding global markets.

2. Extraction Technologies

2.1 Traditional Technologies

Until towards the end of the 20th century separate technologies had to be used to extract essential oils and oleoresins. Thus the extraction of essential oils utilised steam to vapourise the oil, the most common technique being steam distillation extraction (SDE). In the operation of steam distillation, the material to be extracted is charged into a basket, the basket being lifted into the distillation still where it sits on a perforated grid. The top lid is then clamped down and steam from the boiler is passed though the bed of material. The essential oil in the material vapourises and is carried off with the steam into the condenser where the oil/steam mixture condenses. This mixture then passes down into the separator where the oil and water separate by virtue of the immiscibility of the oil in the water, the oil floating on top of the water phase.

Oleoresins however are not volatile, so cannot be separated from the plant materials using steam distillation. They are usually extracted by solvent extraction using a suitable liquid solvent which dissolves the oleoresin but not the matrix of the plant material. Various techniques have been used over the years, but typically the plant material is charged to a vessel containing the solvent e.g. ethanol. After a suitable period of time the vessel is discharged and the solvent with the oleoresin is separated from the spent plant matrix. The oleoresin is then subjected to distillation to separate the oleoresin from the solvent. The solvent is recycled for reuse.

2.2 Supercritical Fluid Extraction

In the last decade of the 20th century, the concept of Supercritical Fluid Extraction was promoted as a viable and more flexible alternative to both Steam Distillation and Solvent Extraction. A typical flow sheet for a three-vessel, two-separator, commercial process is shown in Figure 1. The extraction is effected by passing carbon dioxide at high pressure and just above room temperature through a packed bed of the plant material charged to the extractors. The essential oil/oleoresin dissolves in the supercritical carbon dioxide and passes to one of the separators where the pressure is let down to release the extract. The carbon dioxide is then compressed back to extraction pressure and recycled back through the extractor. It is a batch process so that when operating, two of the units could be extracting whilst the third unit will be discharging and recharging. The extract is taken off the base of the separators.

The critical temperature and pressure for carbon dioxide is 31°C and 72.9 bar. Such units usually operate in the range 30°C to 70°C and up to 500bar pressure. The higher pressures will extract both essential oils and oleoresins as a mixture, but it is however possible to operate the system at just sub critical conditions to extract essential oils only.

![Figure 1. Flowsheet for Commercial Supercritical Fluid Extraction System](image-url)
3. Comparison of SFE with SDE and Solvent Extraction

3.1 Steam Distillation
Steam distillation has been well established as the process for extracting essential oils from plant materials for well over 100 years. The major disadvantages in its use are that it uses an elevated temperature of ~100°C with some potential temperature degradation of the extracted oils, and that distillation times are relatively long, usually up to 8 hours.

3.2 Solvent Extraction
Solvent extraction is a complex process with more processing steps. Since a solvent is used to extract the oleoresin, the solvent/oleoresin mixture has to be separated by fractional distillation. This is not only expensive but it means that the oleoresin product will always be contaminated with the solvent. This can limit its use for food products.

3.3 Supercritical Fluid Extraction
The major advantage of SFE is that it can extract both essential oils and oleoresins, making it much more flexible in a multiproduct environment. The process also operates at temperatures not much above ambient, so that the chemistry of the extracted product is not modified significantly by the process. Extraction times are also shorter than that using the traditional techniques. The major disadvantage is Capital Cost. It is much more expensive than Steam Distillation.

4. Supercritical Fluid Extraction Characteristics
In order to provide background data towards the development of a commercial Supercritical Fluid Extraction industry in Trinidad and Tobago, laboratory scale SFE equipment has been installed and operated in the Extraction Laboratory of the Department of Chemical Engineering at The University of the West Indies (UWI), together with a GCMS to determine the chemical composition of the products from the extractions. The extraction characteristics of a large number of indigenous plant materials have been determined utilising a 100 ml extraction vessel, together with evaluations on the effects of the process variables on the extraction process e.g. Raw material size, Carbon dioxide pressure, temperature and flow rate. Typical results are presented in order to demonstrate the capabilities and flexibility of SFE.

4.1 Oleoresin from seeds/nuts
Commercial products are extracted from seeds and from nuts mainly as cooking or salad oils, but also as health remedies. Pumpkin seed oil for instance is used as a dressing on salads, in marinades, breads, soups and snack foods (Patel, 2013) but is also incorporated into gel-capsules to be used to treat prostate disorders (GNC Holdings, 2014).

A typical extraction curve for pumpkin seeds (Sellier, 2016) carried out at 50°C and 250 bar is shown in Figure 2.

![Figure 2. Extraction Curve for Pumpkin Seeds – Temperature 50°C, Pressure 250bar](image)

Reference to Figure 2 shows that extraction was essentially complete after about 3 hours. The samples were then treated to produce the fatty acid methyl esters (FAME) which were then analysed by way of Gas Chromatography (GC); the same procedure was then applied to a GNC™ pumpkin seed oil sample (GNC Holdings, 2014). Comparison of both chromatograms showed that the SFE oil samples had three major peaks in common with that of the GNC™ oil. While further work is required to identify, and quantify the components, these preliminary investigations suggest that the local oil may have the potential to be of comparable quality to that sold internationally.

4.2 Extraction from flowers
High quality perfumes often utilise the extracts from tropical flowers in their bases. For example, the essential oil extracted from ylang ylang (Cananga odorata forma genuina) is the perfume base for Chanel #5 and is produced on islands in the Indian Ocean utilising steam distillation (The National World, 2015).

The more important flowers which can be grown locally are jasmine (Paltoo, 2002) and ylang ylang (Watson, 2005), for which extensive extraction investigations have been carried out at UWI. A typical series of extraction curves for ylang ylang as carried out by Watson (2005) is shown in Figure 3, with varying operating pressure from 100 bar to 450 bar at a temperature of 45°C. Reference to Figure 3 shows the strong effect of operating pressure, with the quantum of
extraction at 450 bar at 1.5% being almost 3 times that at 100 bar.

The yield from SFE at the higher operating pressures was similar to that from steam distillation. However, the chemical compositions, when compared to Buccellato’s (1982) data, were found to be significantly higher in the less volatile compounds such as benzyl benzoate and benzyl salicylate. Similar observations were made when compared with the compositions reported by Michał Brokl (2013) for the volatile fraction of their extracts. These differences were expected since the SFE extracts’ compositions were being compared to those determined from steam distilled extracts.

4.3 Extraction from Root Crops
A number of tropical species secrete essential oils and oleoresins in their roots. Typical examples are vetiver, where the essential oil is used in perfumery, and the spices turmeric and ginger where the extracts, which contain both essential oils and oleoresins, are used as food additives and for various medicinal treatments. Traditionally in order to extract both the essential oil and the oleoresin, milled turmeric has to be subjected to both steam distillation and solvent extraction. However, a process has been developed at UWI whereby dried ground turmeric is subjected to carbon dioxide extraction at two sets of conditions. Thus, the powder is first contacted with carbon dioxide under subcritical conditions whereby the essential oil is extracted but none of the oleoresin. After all the essential oil is extracted, the pressure is increased and the turmeric is subjected to supercritical fluid extraction, which then extracts the oleoresin.

Figure 4 shows the combined extraction curve where young oven-dried turmeric powder was subjected to 25°C and 65 bar pressure for 1 hour which initially extracted the essential oil, seen to be approximately 5% of the original mass. The temperature and pressure were then increased to 50°C and 200 bar, respectively, for a further 2 hours during which the oleoresin was extracted. The final total extraction was 11.46%. The major components of the essential oil were α-tumerone, curlone, α-tumerone, β-sesquiphellandrine, zingiberene and α-curcumen. The oleoresin normally contained between 50% and 60% curcumin (quantified by UV-VIS Spectroscopy), a compound that showed positive results in the treatment of patients with pre-cancerous changes in different organs; clinical trials of a directly injectable form of curcumin for cancer treatment are currently ongoing (Cancer Research UK, 2015).

4.4 Extraction from Leaves/Fruits
Research on the extraction of essential oils from leaves has been carried out by Maharaj (2011) on basil, and by Clarke (2012) on bay leaves. The results on the extraction of bay oil from bay leaves have been published by McGaw, et al., (2016). In the work a direct comparison of SFE with steam distillation was made for the extraction of the essential oil from Bay (Pimenta racemosa) leaves. It was found that the quality components of bay oil were extracted by SFE in the first hour after which the extracts were increasingly contaminated with higher components and waxes. The maximum yield of the total oil extracted was approximately 4.0% at 40°C and 150 bar pressure. A similar yield was obtained using Steam Distillation but it took twice as long. The SFE extract however had higher concentrations of the quality, i.e. phenolic, components. Thus the eugenol content for the SFE extract was 62.0% compared to 51.4% for the SDE extract, and the chavicol component for the SFE extract was 26.3% compared to 23.0% for the SDE extract. It is appropriate to note that
there is a bay tree plantation in Tobago which can be used to source the raw material.

Another prime candidate for commercial exploitation is that of hot peppers, the oleoresin from which contains capsaicin, used extensively for medicinal purposes for example, in treating arthritis (Arthritis Research UK, 2016), in pepper sprays and as a food flavouring.

Experimental work by Holder (2007) has demonstrated that SFE is ideally suited for producing both the essential oil and the oleoresin.

5. Commercialisation of SFE in Trinidad and Tobago

The market projections for growth in both the essential oil and oleoresins markets over the next 5 to 10 years are buoyant (Grand View Research, 2016a) (Grand View Research, 2016b), demonstrating an opportunity for Trinidad and Tobago to re-enter the business.

The experimental extraction results, detailed in Section 3 for seeds/nuts, flowers, root crops and leaves/fruit, have demonstrated the versatility of SFE as a process to extract valuable products from plant materials which can be grown nationally. The results have also shown that both essential oils and oleoresins can be produced by SFE even during a single run. In addition, the extraction rates are faster than steam distillation and the extract product quality is at least as good as steam distilled products.

It is therefore appropriate to review the prospect for commercialising SFE in Trinidad and Tobago by developing a techno-economic feasibility study. It is clear from the experimental results that technically the process has the flexibility to produce from a wide variety of materials at different times. In addition, when using a 3-extractor system as shown in Figure 1, it is even possible to extract from two materials at one time.

A feasibility study has therefore been carried out to see if such an operation could be economically viable. In carrying out the study, a 3-extractor system, similar to that shown in Figure 1, was considered, and to simplify the exercise a single raw material was assumed; that being turmeric.

Such systems are limited in size because of the operating pressure. A quotation for a 3 by 1,000 L extractor system utilising 2 separators was obtained from the major supplier of such systems in the USA, the supply of a skid mounted system being given as ~US$6.5m. Recognising that the turmeric has to be dried and milled prior to processing and that a building for raw material storage, processing, product store, laboratory and administration had to be built, the overall capital cost was estimated to be ~US$7.5 millions.

Supply of raw material was assumed to be from contract farmers at a factory gate price of US$0.60 per kg. The preferred method however would be to produce on land next to the factory site using mechanical methods to reduce cost.

Assuming that the plant would operate for 310 days in the year, a plant utilisation factor of 85% with a batch turnaround time (filling, extracting, discharging, recharging and down-time) of 3 hours it is estimated that 148,800 kg of extract would be produced annually. The current global price for turmeric extract is US$50 per kg, so the estimated annual revenue is US$7.44m.

With an annual operating cost estimated to be US$4.27m, the Internal Rate of Return over a 5-year projection was calculated to be 21%, thereby indicating that such a business could be financially viable. Carbon dioxide is readily available in Trinidad as a by-product of ammonia manufacture.

It was assumed in the feasibility that the extract would be marketed without further processing, and sold through brokers in the USA and Europe. It is envisaged that this would only be the first step in the development of the industry. The second stage would be the separation of the valuable components of the initial extract by fractional distillation. The valuable components could then be sold directly to the manufacturers of the consumer products at significantly higher prices. The third stage would be the development of specific consumer products for marketing locally, regionally and globally, thereby benefitting fully in the value added chain.

5. Conclusion

It may be concluded that the introduction of a Supercritical Fluid Extraction Industry in Trinidad and Tobago would be viable technically and could also be financially viable. It is recommended that a complete Business Plan be developed and investors sought for the enterprise.
Garlic, Onion), By Application (Food and Beverage, Flavors, Pharmaceuticals) and Segment Forecasts to 2024, Grand View Research, Inc.


Holder, R., (2007), Processing Potential of the Capsicum Genus, Ph.D Thesis in Food Technology (Unpublished), Department of Chemical Engineering, The University of the West Indies, Trinidad and Tobago

Maharaj, S. (2011), Extraction Studies with Basil, Ph.D in Chemical Engineering (Unpublished), Department of Chemical Engineering, The University of the West Indies, Trinidad and Tobago


Paltoo, V. (2002), Supercritical Fluid Extraction of Jasmine, Ph.D in Chemical Engineering (Unpublished), Department of Chemical Engineering, The University of the West Indies, Trinidad and Tobago.


Sellier, B. (2016), A Process for the Supercritical Fluid Extraction of Pumpkin Seed, M.Sc. Dissertation (Unpublished), Department of Chemical Engineering, The University of the West Indies, Trinidad and Tobago.


Watson, M. (2005), Supercritical Fluid Extraction of Ylang Ylang, PhD in Chemical Engineering, (Unpublished), Department of Chemical Engineering, The University of the West Indies, Trinidad and Tobago.

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Sharad Maharaj is Lecturer in Chemical Engineering at The University of the West Indies, where he specialises in Separation Processes and Numerical Methods. Dr. Maharaj holds a Ph.D in Chemical Engineering from The University of the West Indies (UWI) in the area of extraction processes with special reference to the use of Steam Distillation and Supercritical Fluid Extraction to extract high value products from indigenous plant materials. He currently leads the research work of the Extraction Laboratory at The UWI.

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Rosemarie Skeene is Senior Instructor at The University of Trinidad and Tobago. Mrs. Skeene holds a Master of Philosophy degree in Chemical Engineering and a Bachelor of Science degree in Chemistry and Zoology. At present, she is pursuing her PhD in Chemical Engineering; her research is on the Extraction of Turmeric using Sub-critical and Supercritical CO₂.

David R. McGaw holds B.Sc and M.Sc degrees from The University of Wales and a Ph.D from The University of the West Indies. After working as a Process Engineer in artificial fibers and subsequently in compound fertilisers, he joined the teaching staff of The University of the West Indies in 1967. He retired in 2003, having also served as the Head of Department and Dean of the Faculty of Engineering. In 2006, he joined The University of Trinidad and Tobago (UTT) as Vice Provost, before being appointed Provost in 2007, retiring from the UTT in 2009.