PART 4 – AROMA CHEMICALS DERIVED FROM ESSENTIAL OILS

9 TECHNOLOGY, INTELLECTUAL PROPERTY, RESEARCH AND DEVELOPMENT

9.1 Standard Technologies

There are several options available for the "extraction" of essential oils from plant material. By far the most common locally (and internationally) is steam distillation. All the crops in Lists A and B of Appendix "D" can be distilled by steam distillation.

In the steam distillation process, steam is generated in a boiler and is piped into the bottom of the still vessel which contains the plant material. As the steam rises through the plant material the volatile essential oils are released and are carried upwards with the steam and pass from the top of the still through a condenser. The condenser is continually cooled by cold water passing through a system of internal tubing. This cools the steam (water and oil mix) into a liquid. The water and oil mixture flow into a separator (usually a Florentine flask) where the oil, generally having a specific gravity less than water, floats to the surface and is collected. The water is collected separately as "hydrolats" and the oil is further "dried" to remove any excess water.

The oil is then bottled in opaque jars (preferably glass or aluminium) and kept at cool temperatures. Most oils suffer a degradation of quality if they are exposed to sunlight or high temperatures.

The key components of a steam distilling unit are:

- The boiler to generate steam ;
- The still for holding the plant material to be steamed;
- The condenser for distilling the steam into oil and water; and
- The separator (usually a Florentine flask) used to separate the water from the oil.

Although the technology is reasonably simple the distillation process needs to be well-managed and there are several factors that can impact on the yield and quality of the oil:

- Besides the harvesting issues (such as time of harvest), there are the post harvest treatment of materials which varies from crop to crop. Some crops benefit from a little drying as this reduces the water content and thereby exposing the oil glands more effectively. Other materials need to be distilled fresh as they quickly degrade and loose their volatile components.

- The heat and pressure under which distillation occurs is important as it can effect yields and the composition of the oil. It is possible to overheat or burn oils and thereby change their composition.
- The time of distillation impacts on cost of production. The efficiency curve for oil extraction implies that a distillation time beyond one hour is inefficient.
- Proper packing of plant material is important to ensure that the steam rises uniformly through the charge. This affects yields.
- A good head of steam and good flow rate are required to achieve good yields.

Accordingly, distillation needs supervision and the parameters need to be measured and matched against the analysis of batches of oil, which then provides feedback on the success of the distillation process.

There is a lot of literature and opinion concerning still design and operation[68]. Detailed information can also be acquired from reputable sources like Agrelek (who provide technical support to those using electricity for agricultural purposes) and those companies that manufacture stills and related equipment.

9.2 Capital and Operating Costs

Costs of a standard distillation unit with a one ton still, with boiler and condenser is approximately R500,000. There are cheaper options but these are not advised. The cost is not a result of the components being imported (they are not), but because they are best made out of stainless steel. The stills are made locally by engineering works. The other components the boiler and the condenser and Florentine flasks are purchased “off the shelf”. There is also an occasional secondhand market for distillation equipment. In some markets, in order to upgrade oil quality and returns to the growers, governments have introduced cheap but effective stills (e.g. Sri Lanka, Indonesia and Bhutan). Consideration could be given to research into a “government issue” still and whether or not this would provide any economic benefits.

[68] See for example Appendix “F” an interesting extract from a consulting report done by CL Green for the Cambodian Government

The running cost of distillation (besides cost of capital) is the labour and the power for heating the boiler. Fuel sources for the boiler include:

- Coal or timber – cheapest if readily available (e.g. Nelspruit region)
- Diesel – expensive therefore needing larger still unit to process economically
- Electricity – is similar to coal provided one is able to process outside the peek tariff times (if not the cost is more equivalent to Diesel).

The distillation costs (excluding capital costs) only make up some 1% of the cost of producing the oil. If one includes the capital costs this rises to almost 30% for a 25 hectare operation. However, once the producer scales up to 50 hectares this percentage drops to around 10%. The estimated cost per charge (one ton still) is R120.[69]

9.3 New extraction methods

All methods of distillation can be performed at above or below atmospheric pressure and this can impact on the composition of the oil. Some oils are deliberately extracted in a vacuum so as to enhance the occurrence of certain chemical compounds. These technologies are used in the secondary beneficiation processes, where further distillation under varying temperatures and pressures, allows for the isolation of selected components of the oil.

In mature oils markets (such as the citrus oil industry) the modern extraction plants are fully computerized and can produce oils whose composition is accurately controlled and which conform to pre-determined standards and end user requirements[70].

Carbon Dioxide-extracted essential oils were conceived during the search for a complete replication of nature by extracting without damage or adulteration. The heat involved in normal extraction does effect the composition of the essential oil. CO_2 when at sub-ambient temperatures is liquid and acts as a natural solvent.

The critical point for CO_2 is at 31°C and at pressure 73.8 bar. Sub-critical Liquefied CO_2 (between -55 and 31 °C and 5 to 74 bar) behaves as a non-polar solvent. CO_2 operating in the normal working conditions, between 0 and 10 °C and 50 to 80 bar, acts as a selective solvent. In this range, CO_2 has the density of fluids, low viscosity and the diffusion properties of gas. By varying the temperature and pressure within a range one can be selective about the substrate of extraction. As a solvent, CO_2 is odourless, tasteless, colourless and easy

[69] Parameters provided by Biosys Plant Extracts (Pty) Limited
[70] Swaine and Swaine (1988) Citrus oils: processing, technology and applications. Perfume & Flavourist 13(6), 1-20

to remove. CO_2 is also readily available, cheap and easy to recycle, making operational costs cheap. However the initial equipment cost is quite high. Since the equipment operates under controlled pressures it must be engineered from high quality stainless steel and all welds are x-ray tested. Such equipment can be manufactured in South Africa. A small scale commercial plant is estimated to cost R1.5 million.

Due to the relatively low temperature of CO_2 extraction, the extracts are different from steam distilled oils. The liquid CO_2 extracts also include light fractions of resins, giving the extract and element of an "absolute". In this manner the liquid CO_2 extract is more representative of the original botanical material. Although this is attractive, it does introduce change to the end result and the users of the products in the main are still to be convinced of the relative benefit of the "improved" extracts in the light of the additional costs.

The main benefits of CO_2 extraction may be summarized as follows[71]:

- No solvent residues. Extracts prepared with organic solvents are heated under vacuum to remove the residual solvent. This heating can negatively impact the top notes which evaporate. In some cases to preserve the top notes a residue of solvent is retained.

- No "off notes". During steam distillation the heat causes decomposition of certain materials and generates traces of nitrogen and sulphur compounds. These give unpleasant "still notes". Although these still notes sometimes age and disappear if the oil is kept, this required storage for up to 6 months. CO_2 extracted oils have no such still notes.

- More "top notes". Due to the lower operating temperature the volatile top notes are preserved.

- More "back notes". Since CO_2 extracts the character of the "absolute" – molecular weight 200-400, these components add to the overall character of the extract.

Research on the technology began in the 1960's and reached its peek in the 1980's. The outstanding task for today's researcher is to apply the technology to actual materials and to document the effects and benefits of the technology to industry. Even worldwide, the technology is not used for many essential oil products and in fact is used mostly in specialized food technology applications, such as removing caffeine from coffee beans.

[71] David A Moyler, Liquid CO2 extraction in the flavour and fragrance industries ,Chemistry and Industry, 17 October 1988

In South Africa, the use of the technology is still mostly at laboratory or pilot scale. For example, the University of the North West (formerly, Potchefstroom University) is involved in projects involving Chamomile, Artemesia, Lemon balm and Tswane University of Technology is involved in extracting oils such as Avocado oils. Only Afriplex (Pty) Limited, in the Cape, appears to be using it on a commercial scale (Rooibos extraction). Generally it is considered too expensive and the benefits are not yet demonstrable at the cost.

9.4 Intellectual Property Issues

In respect of the production of essential oils, the intellectual property issues fall into three categories:

- Agronomic
- Distillation
- Market Knowledge

The intellectual property issues are more a matter of trade secrets and are not of the nature of constraints relating to patented processes. On the whole there is a lot of general published information available on crops (even technical information on agronomy and oil composition). With regards to the agronomy the intellectual property is more about lessons learned in the field. Even with regards to distillation, the state of the art is public knowledge and has been for many years. There may be some registered designs where persons have innovated around layout and design. However, the engineering firms that manufacture the stills locally are qualified and experienced in design of this equipment. The other key technology is the steam boiler. This too is old technology and suppliers are able to provide advice.

The true intellectual property in the supply chain rests predominantly in the market knowledge and the ability to interact with Flavour and Fragrance houses. Although this knowledge is not proprietary it is difficult to acquire. To be successful one needs chemistry and organoleptic skills and experience in order to analyze oils and match them to prospective buyers. The essential oils industry, as well other flavour and fragrance segments, requires a unique combination of science and art. Taste and smell are complex senses and different cultures and markets have different requirements. Since the market is “sample” driven it is important to know where to send the particular oil to. Successful players in the market develop a “library” of essential oils and the requirements of the various markets.

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Furthermore, there is some scope for the chemist to “rectify” an oil to make it more acceptable. This of course increases profitability.

The area where intellectual property rights issues may arise is the area of the exploitation of indigenous plants, where use is made of indigenous knowledge to arrive at commercial applications. This matter came to the South African public’s attention in the case of the appetite suppressant drug P57 which was derived using the indigenous knowledge of the Khoi people. The developers of P57 were obliged to recognise the interests of the indigenous people. It is now recognised that indigenous knowledge of a plant and its uses represents a significant step and a cost saving in the process of bringing a new product to the world market. At present no legislation exists to cover these issues, but a proposed law to be known as the "Protection of Indigenous Knowledge Act" is being prepared to advance the promotion and protection of indigenous knowledge.[72] The purpose of the act is not to prohibit the exploitation of indigenous plants but seeks to promote and develop the use of indigenous genetic material, by ensuring that the rights of the lawful owners of the initial knowledge are taken into consideration as the plant material is commercially exploited. It is hoped that the proposed legislation will contribute to documenting indigenous knowledge and to providing a platform for commercialisation. The key to the legislation’s success will be whether or not it can provide a clear mechanism for the creation of legally defensible intellectual rights and a mechanism for licensing these rights to commercial entities for commercialisation.

9.5 Research & Development

The potential for research and development falls into several categories. For the purposes of this Study we are not specifically considering the body of research that has to be undertaken or continued with regards to the agronomy of essential oil production, however this is fundamental and cannot be totally ignored. This category would include matters such as:

- The selection of correct plant types for propagation;
- The investigation of pathogens and pests related to each plant species;
- The identification of “organic” methods of pest control and fertilization;
- The investigation of agricultural best practice with regards to planting, cultivating and harvesting

[72] Coetzee, C., E. Jefthas, and E. Reinten. 1999. Indigenous plant genetic resources of South Africa. p. 160–163. In: J. Janick (ed.), Perspectives on new crops and new uses. ASHS Press, Alexandria, VA.

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With regards to the fundamentals of essential oil production (and therefore excluding R&D related to Flavour and Fragrance applications and formulations), the following R&D needs have been identified:

- The development of distillation technologies that are best suited, both technically and economically to the South African environment.
- The development of methods of infield testing (e.g. to measure essential oil qualities in the field, useful in determining the best times to harvest plant material).
- The development of a body of knowledge around South African indigenous plants, as they relate to the F&F industry (particularly information relating to toxicity and allergens).

The first category concerns the development of standard distillation unit designs that suite South African conditions, mostly with regards to fuel sources and their method of use in the field. As has been stated previously the technology is public knowledge. However, there has been some learning with regards to dimension and configuration of units. The cost issue also needs some attention. Most stills have been built to user specification by an engineering works. Unless there is a larger demand for units it is unlikely that anyone will try and improve the still production process and reduce costs. This could be an area for some R&D spending in conjunction with a reputable engineering works and industry players. Another area is the design and production of mobile or portable stills. Various industry players (e.g. Teubes and Biosys) are considering this because the initial cost of a distillation unit is a barrier to introducing a new producer to the essential oils industry. The economics of such a mobile unit require that it be quite large and therefore requires significant innovation to optimize the design. In addition, the Consultant was advised that there was a minor issue which may be of some significance in projects where there is water scarcity. The condensing process relies on cold water. Unless the cold water is chilled it cannot be recycled and is disposed of. Some technically advanced installations have chillers and the water is recycled, alternative use is made of cooling towers. On some projects the warm water is simply disposed of and fresh cold water is used. This may not always be economically or environmentally appropriate. This matter may require some research.

The second category is concerned with addressing the problem that farmers face in the field. With most other crops the farmer can assess progress by physical examination of the plant or fruit. With essential oils there can be a marked difference in the yield and composition of the oil from one season to the next. Moisture, daylight and temperature can all significantly impact on the important components of the oil. International Industry expert, Brian Lawrence (who has a regular column in the Perfumer & Flavorist) routinely publishes comparisons between various oils which show the huge variances in characteristics, often within the same crop but under different conditions or time of year. Since the quality of the oil is the farmer's main concern it would be helpful if ways could be found to give the farmer the ability to check the presence of certain of the key chemical components in the field. The process of getting a full laboratory analysis is costly and time consuming.

The third category is the most important of all, particularly if the end objective is to exploit the indigenous crops internationally. There has been a recent spate of legislation in the major international markets, which is aimed at regulating the inclusion of chemicals in food stuffs and other products used by people. This has been widely reported on[73]. As an example, the European Union (South Africa's largest trading partner for most essential oil products) has introduced labelling requirements that have caused large manufacturers to insist that the Flavour & Fragrance houses only include permissible ingredients in their formulations. This has introduced an additional element of conservatism into the Flavour & Fragrance industry. The main issues are toxicity and allergens. The majority of additives (be they flavors, colors or fragrances) are listed as to whether they are GRAS (Generally Regarded As Safe) or not. The process of GRAS is more or less like the process negating toxicity in clinical trials for pharmaceuticals. In addition, the EU has identified 26 allergens, 16 of which are present in essential oil products. A list of the essential oils and their percentage content of allergens is attached hereto as Appendix "E". Other key standards are those set by FEMA, which was established by industry in the USA to promote self-regulation in respect of additives used by food manufacturers. In the light of the above and in order to graduate from a raw material supplier to a knowledge based industry, it is necessary for South Africa to develop skills and knowledge concerning the international standards and to have its own processes for investigating and analyzing the chemical composition of local plant extracts and materials destined for Flavor and Fragrance application, particularly where this may be destined for the international markets. If these skills are not developed South Africa will remain a mere producer of raw materials, as opposed to being a quality supplier of flavour and fragrance compounds. A proactive approach would give local producers a platform for negotiations and provide buyers with added confidence in the industry. This approach would generally help South Africa establish itself as a serious player in the essential oils market.

9.6 International R&D - Case Study

The Australians are known for the deliberate manner in which they tackle development matters. Their treatment of the essential oils industry is no exception. Essential oils is a relatively new industry for Australia (other than eucalyptus oils). The Australian Rural

[73] Perfumer& Flavorist March/April 2004

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Industries Research & Development Corporation (RIRDC)[74] is a Government organization that promotes rural industry in Australia. In 2002, the RIRDC published its R&D Plan for Essential Oils and Plant Extracts for the period 2002-2006. The Essential Oils and Plant Extracts Programme has been running since 1990 and for the period 1990 to 2001 some A$ 11 million has been spent on about 70 projects falling into five research categories:

- Production (industry competitiveness – agronomy)
- Processing (value addition – extraction and processing)
- Training and development (encouraging associations, workshops etc)
- Communications and technology transfer (promoting adoption of research findings raising the general standard of industry through shared information)
- Markets (gathering market information and promoting sales and recognition of quality)

The content of the RIRDC Plan and the fact that it exists at all is of interest. The previous plan had dealt with the issues of:

- Increasing productivity (through plant breeding, agronomy and plant protection)
- Improving harvesting and post-harvest handling, and extraction
- Establishing a stable grower base
- Implementing quality assurance and control programs.

The priorities identified for the next period (i.e. for 2002-2006) were:

- Improved market information (particularly future trends)
- Improved products (encourage development and distribution of improved genetic material)
- Improved production systems (especially pest/weed/disease control)
- Regulatory approvals (regarding indigenous products, assist registration through organizations such as FEMA, by providing research information)
- Improved post harvest treatment of plant material (improve quality by sophisticated extraction and treatment so as to differentiate from low cost competitors)
- Develop the industry (overcome effects of fragmentation by supporting growers organizations, improved communication and setting of national standards)

74 www.rirdc.gov.au

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The RIRDC had originally focused on some seven essential oil crops but has generated a secondary list of some twenty more that are now to receive more attention (interestingly the secondary list includes Buchu).

The content of the RIRDC Essential Oils and Plant Extracts Program illustrates that the issues facing the South African industry are similar to those faced in other nations. What is challenging is the concerted effort that some nations put behind fledgling industries, not necessarily by way of subsidies or price protection but by helping create capacity, knowledge and competitive advantage. One of the key features of the RIRDC Program is the policy that there is full disclosure and dissemination of research findings, with the expressed objective of raising the overall knowledge-base of the local industry. It is suggested that South Africa adopt this type of approach to attractive industries such as the essential oils industry and other non-food crops.