TECHNICAL NOTE

Extraction kinetics of essential oils from nine sequential fractions of 100 g lemon peels, each extracted for two hours using the Naviglio® Extractor and the same initial solvent.

Sequential extraction kinetics of essential oils from lemon peels (Citrus limon) using Naviglio® technology

1. Objective

This note describes the extraction process of essential oils from lemon peels (Citrus limon) using Naviglio technology, based on experimental trials carried out on nine 100 g fractions of fresh matrix, each extracted for two hours with the same initial 99% (v/v) ethanol. This solid–liquid extraction protocol allows the study of the extraction kinetics of lemon essential oil from lemon peels and the determination of the solid-to-liquid ratio relative to saturation. It also highlights the high extraction efficiency of Naviglio technology. Finally, the resulting alcoholic extracts, with increasing concentrations of lemon essential oil, can be used for various industrial applications, ranging from the beverage industry to cosmetics and perfumery.

2. Principle of Naviglio technology

The Naviglio extractor operates on the principle of **dynamic solid–liquid extraction under controlled pressure**, based on the alternation of:

- **Static phase**: the system is brought to a preset pressure, promoting the penetration of the solvent into the pores of the plant matrix;
- **Dynamic phase:** sudden depressurization generates a pressure gradient between the interior and exterior of the matrix, inducing the forced release of extractable compounds.

This mechanism does not rely on passive diffusion but on a mechanical extraction effect, which "activates" the extraction process, as stated in the Naviglio principle. 1

Thanks to this mechanism, solid–liquid extraction is completed in significantly shorter times compared to traditional methods such as maceration (infusion) or percolation. Furthermore, extraction can be carried out at ambient or sub-ambient temperatures, since the process does not depend on the diffusion of active compounds. In general, neither the solid matrix nor the extracted active compounds undergo degradation, as the solid–liquid contact is limited to durations ranging from a minimum of two hours to a maximum of 24 hours for more resilient matrices such as barks, roots, and berries. Naviglio technology allows operation at room temperature, preserving sensitive monoterpenes such as limonene, β-pinene, and γ-terpinene. The absence of prolonged heating minimizes oxidative phenomena and prevents alterations of the aromatic profile.

This technical note demonstrates the capabilities of the Naviglio Extractor (Atlas Filtri, Limena, PD, Italy – www.atlasfiltri.com) in rapidly and reproducibly extracting lemon essential oil, using absolute ethanol as the carrier solvent (99.9% (v/v) Alcoolital, Fossano, CN, Italy). This fundamental experiment on rapid dynamic solid–liquid extraction cannot be performed with any other currently known solid–liquid extraction technique.

3. Experimental Procedure for the Extraction of Lemon Peels Using the Naviglio Extractor

Materials and Equipment Used

20 kg of lemons, 99.9% (v/v) ethanol (Alcoolital, Fossano, CN, Italy); laboratory oven, analytical balance, 0.45 µm PTFE syringe filter (Millipore), 10 ml pipette, Naviglio® Extractor mod. 500 cc (Atlas Filtri, Limena, PD, Italy).

A batch of approximately 20 kg of fresh organic lemons from the Phlegraean area of Pozzuoli was processed over three separate days as follows:

Each day, 3 kg were selected from the 20 kg batch, ensuring that the peels were free of visible defects (e.g., black spots, mold). The selected lemons were quickly washed with spring water and dried with absorbent paper. Then, 1 kg was peeled using a citrus peeler, taking care to minimize inclusion of the albedo, which contains no essential oil and would introduce an error in the weight of the flavedo (the yellow part containing the oil cells).

One hundred grams of peels, obtained from 1 kg of lemons, were placed into a 100-micron food-grade polyethylene filter bag, which was then inserted into the chamber of the Naviglio Extractor mod. 500 cc (Atlas Filtri, Limena, Padua, Italy; www.atlasfiltri.com). The solvent used for the extraction of lemon essential oils was 99.9% (v/v) ethanol (absolute ethanol) (Alcoolital, Cuneo, Italy) in a volume of 550 ml.

The operational conditions were as follows: static phase: 2 minutes; dynamic phase: 2 minutes with a 12-second piston pause, for a total of 15 cycles, corresponding to one hour of extraction at room temperature (18–20 °C) and at the maximum working pressure of 10 bar.

At the end of the first hour, the ethanol was completely discharged from the extractor and homogenized by agitation in a 1-liter glass bottle. An aliquot of 12 ml was filtered through a 0.45 µm PTFE filter. Exactly 10 ml of the extract were transferred using a double-marked glass pipette into a 25 ml beaker (previously brought to constant weight in an oven at 105 °C on an analytical balance), which was then placed in an oven at 105 °C for drying over 24 hours. After 24 hours, the beaker was allowed to cool to room temperature in a desiccator and weighed on an analytical balance.

The recovered extract was reintroduced into the extractor chamber, and the extraction phase was repeated for another hour under the same operational conditions. At the end of the second hour, the extract was collected, and a second 10 ml sample was taken following the procedure described above.

After the second hour, the first 100 g of exhausted peels were replaced with a new 100 g portion of fresh peels from the second kg of lemons, peeled according to the precautions previously described. The extract obtained from the first extraction process was used as the extracting solvent for this second portion. Since the removal of the 12 ml aliquot reduces the total volume and affects the achievement of the set maximum pressure, an equivalent amount of 99.9% (v/v) ethanol was added to the extract to restore the initial volume of 550 ml.

The second portion of lemon peels was extracted in the same way as the first, and two additional 10 ml samples were collected during the third and fourth hours of extraction. Subsequently, a third portion of lemon peels, obtained from the third kg of lemons according to the procedure described above, was subjected to another two hours of extraction using the extract obtained from the fourth hour (i.e., from the second extraction process) as the extracting solvent. Two further 10 ml extract samples were collected and dried at 105 °C for 24 hours.

At the end of the extraction of the third portion of lemon peels, the extract was stored in a refrigerator at 4 °C until use the following day, while the filter bag was left in the extractor chamber, which was hermetically sealed until the next day. Neither the chamber nor the bag was washed to avoid altering the extraction conditions for the subsequent day.

The extraction processes were repeated over the following two days, carefully following all the steps described above. At the end of the three extractions on the second day, six additional extracts were obtained (seventh and eighth hour; ninth and tenth hour; eleventh and twelfth hour), and the final extract of the second day was stored in a refrigerator at 4 °C until use the next day. Similarly, the filter bag in the extraction chamber was stored without washing.

Finally, on the third day of extraction, samples corresponding to the last six hours of extraction from the final processes were collected.

4. Extraction kinetics

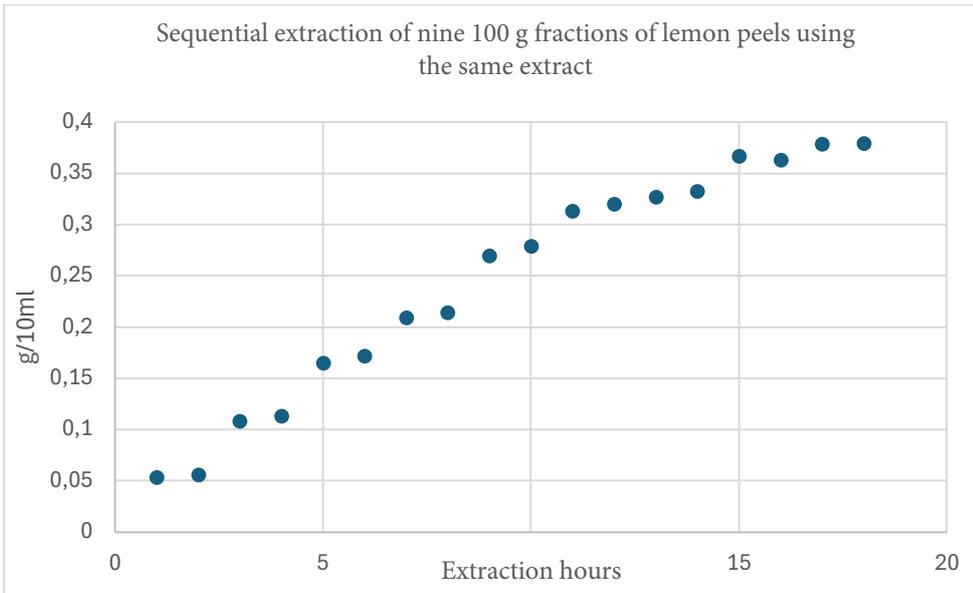**Sequential extraction of nine 100 g fractions of lemon peels using the same extract**

Fig. 1 – Extraction kinetics of nine sequential 100 g lemon peel aliquots, each extracted using the extract obtained from the preceding extraction.
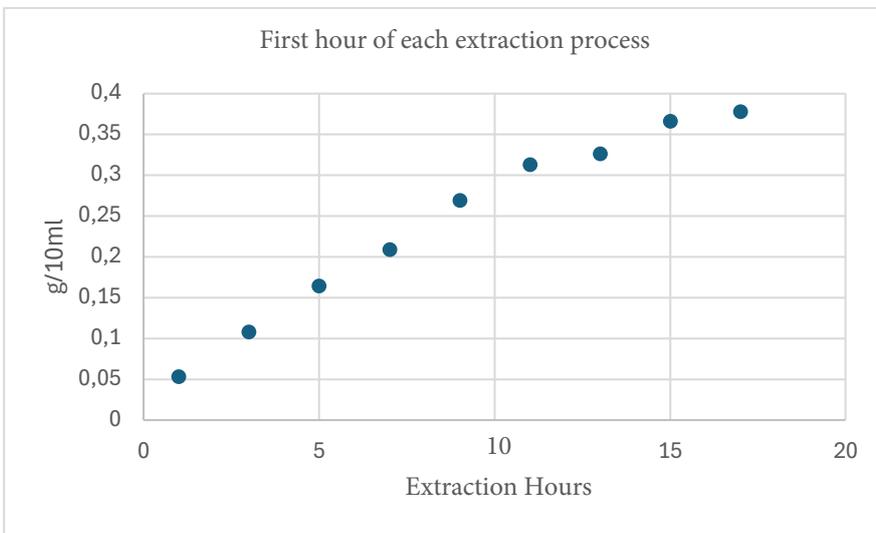
First hour of each extraction process

Fig. 2 – Extraction kinetics of nine sequential 100 g lemon peel aliquots, each extracted using the extract obtained from the preceding extraction. Data refer to the first hour of extraction for each process.
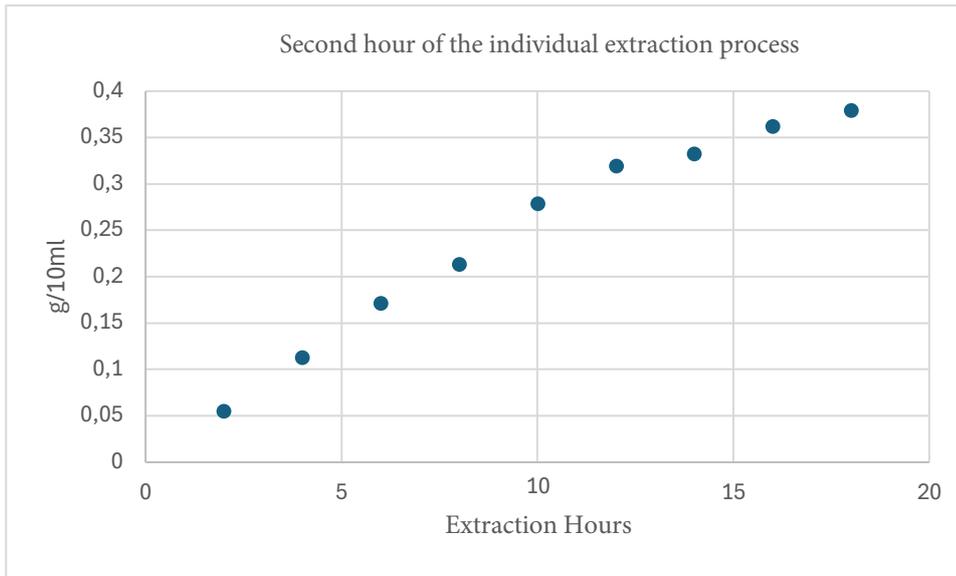
Fig. 3 – Extraction kinetics of nine sequential 100 g lemon peel aliquots, each extracted using the extract obtained from the preceding extraction. Data refer to the second hour of extraction for each process.

Figure 1 shows the overall extraction kinetics related to the nine 100 g lemon peel aliquots extracted sequentially using the same progressively enriched solvent. Figures 2 and 3 report, respectively, the trend of the first and second hour of extraction for each process.

For each aliquot, the extraction kinetics exhibits the typical behavior of solid–liquid extraction processes and is well described by a negative exponential equation:

$$C = S \, e^{-k/t}$$

C represents the concentration of the extracted compounds, S the saturation concentration, and k the kinetic constant.

The experimental data indicate that two hours of extraction are sufficient for the complete exhaustion of essential oils from the lemon peels. This behavior is consistent across all analyzed aliquots, demonstrating the high reproducibility of the process.
The trend of the graph confirms efficient extraction kinetics during the initial stages, followed by a plateau that identifies the recovery limit under the adopted operating conditions.

Un risultato di particolare interesse è rappresentato dalla linearità della curva di arricchimento del solvente nelle prime sei aliquote sequenziali. In questo intervallo, l'incremento della quantità di composti estratti risulta pressoché proporzionale alla massa di matrice trattata, indicando che il potere estrattivo del solvente rimane sostanzialmente invariato nonostante il progressivo aumento della concentrazione di oli essenziali.

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Overall, 600 g of lemon peels were successfully extracted using 550 mL of ethanol, without any evidence of early solvent saturation phenomena.

The extraction kinetics for each 100 g aliquot of peels is typical of solid–liquid extraction processes and can be fitted with a negative exponential equation $(C = S\,e^{-k/t})$; where C represents the concentration of essential oil in the extracting solvent, S is the saturation value, which in this case corresponds to the complete extraction of essential oils from the individual lemon peel aliquot; k is the process rate constant, and t is time. As shown in the graph, two hours are sufficient to completely extract the essential oils from the lemon peels. This extraction time was deliberately selected as the minimum time required to demonstrate the efficiency of lemon liqueur production, more commonly known as limoncello. A distinctive feature of the studied phenomenon—namely, the sequential extraction of nine 100 g lemon peel aliquots—is that the first six aliquots are described by a linear trend with a correlation coefficient very close to unity. This indicates that the extraction kinetics of the peels is perfectly superimposable.

This result is attributable to the extraction rate achieved by the Naviglio Extractor, which does not induce any degradation of the solid material under extraction and is based on a pressure–depression effect generated by the extracting solvent.

The linearity of the phenomenon demonstrates that there are no appreciable variations in the extraction capacity of the progressively enriched extract toward the lemon peels. The extraction remains linear up to the sixth aliquot, making it possible to achieve a cumulative extraction of 600 g of peels in 550 mL of ethanol—a result that cannot be reproduced by any conventional extraction technique. In particular, in maceration processes, the maximum ratio allowing complete coverage of the peels with ethanol is approximately 150 g per 500 mL. Moreover, achieving a similar enrichment of ethanol through repeated maceration steps would be impractical, as reaching a ratio of 600 g per 550 mL would require approximately 12 weeks under a conventional extraction protocol.

The resulting graph (essential oil yield or concentration of volatile compounds as a function of cycles/time) shows a typical behavior of extraction using Naviglio technology:

Very rapid initial phase: a high release of essential oils occurs during the first cycles, resulting from the removal of easily accessible compounds in the superficial oil glands of the flavedo, as indicated by the high value of the kinetic constant k

Intermediate phase: progressive decrease in the extraction rate, associated with the depletion of the most readily available fraction.

Plateau phase: stabilization of the curve, indicative of the attainment of equilibrium and the near-complete extraction of the compounds under these operating conditions.

The first six 100 g aliquots exhibit a nearly identical trend, indicating **good process repeatability** and matrix uniformity, which, despite its natural origin, demonstrates an exceptionally high homogeneity in the distribution of lemon essential oils within the flavedo.

5. Applications of the extracts

This fundamental solid–liquid extraction experiment not only allows the study of the extraction kinetics of lemon peels and the determination of the process rate and the saturation of the extracting solvent with respect to the extracted matrix (in this case, lemon peels), but also enables the industrial production of extracts that can be used differently depending on their concentration. Specifically, the first two hours of extraction yield the classical extract used for limoncello production; the following two hours produce an alcoholic extract that can still be used for lemon liqueur or as a non-concentrated flavoring (e.g., for soaking cakes); the third extraction produces a concentrated extract that can serve as a base lemon aroma, even in cosmetics; the subsequent three extractions further concentrate the lemon essential oils in ethanol and can therefore be employed in cosmetic applications and as bases for perfumes.

6. Robustness of Atlas Filtri Naviglio equipment

The experiment was carried out using a 500 cc Naviglio extractor, model built in 2002; the remarkable reproducibility of the obtained data confirms the robustness and precision of the equipment in repeating both static and dynamic extraction phases, even after approximately 25 years of use.

7. Conclusions

This fundamental rapid dynamic solid–liquid extraction (RSLDE) experiment, carried out using Naviglio technology, represents a process that can be applied to any plant matrix using any traditional carrier solvent, such as water, ethanol, glycerol, etc. The speed of Naviglio extraction is highlighted by a high value of the kinetic constant, as already demonstrated for various applications reported in the literature. Regarding the extraction of essential oil from lemon peels, the process can be considered complete after two hours of extraction under the operational conditions described in the experimental procedure of this technical note. The progressive enrichment of ethanol with essential oil proceeds linearly up to the sixth extraction of repeated 100 g aliquots of peels.

The enrichment procedure for lemon essential oil can be advantageously used for the rapid quality control of lemons in this case, and more generally for any plant matrix containing extractable active compounds. Concentrating essential oils at room temperature prevents their degradation and allows the production of concentrated extracts in a short time for various industrial applications, including the beverage industry, dietary supplements, cosmetics, and perfumery. Finally, the extraction of essential oils from lemon peels using a Naviglio extractor proves to be rapid, reproducible, and also suitable for the valorization of agro-industrial by-products, as extensively demonstrated in the literature.

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