PAPER

# CHEMICAL AND PHYSICO-CHEMICAL PARAMETERS AND COMPOSITION OF THE AROMATIC FRACTION OF LIMONCELLO

## PARAMETRI CHIMICI E CHIMICO-FISICI E COMPOSIZIONE DELLA FRAZIONE AROMATICA DEL LIMONCELLO

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ABSTRACT

Recently limoncello, an Italian liqueur made with lemons, has gained great popularity with rapid and unexpected diffusion. Characterisation of the product is therefore necessary so that quality and authenticity parameters can be established. The literature does not report any data on the analysis of this liqueur. Hence, in this work, in 13 samples of limoncello, chemical, physico-chemical parameters and composi-

RIASSUNTO

Il trend marcatamente positivo che ha caratterizzato, nell'ultimo decennio, il mercato del limoncello ha suggerito l'opportunità di procedere alla caratterizzazione del prodotto per stabilire i parametri di qualità e genuinità anche in considerazione dell'assenza, in letteratura, di risultati relativi all'analisi di questo liquore. In questo lavoro sono stati analizzati 13 campioni di limoncello. Su tutti i campioni sono stati de-

- Key words: aromatic fraction, gas chromatography, lemon, limoncello, flavour -

tion of the aromatic fraction were determined. The chemical and physicochemical parameters differed considerably from sample to sample. This is probably due to the different cultivars of lemons, pedoclimatic conditions and cultivation techniques, as well as to the different procedures for preparing the liqueur. Results showed that the aromatic components present in the lemon peel undergo many changes during preparation of the liqueur and during storage, mainly catalysed by aqueous acidic medium, high temperatures and UV irradiation.

terminati alcuni parametri chimici e chimico-fisici, ed è stata determinata la composizione qualitativa e quantitativa della frazione aromatica mediante analisi gascromatografica. I risultati ottenuti mostrano che c'è una grande variabilità nei valori dei parametri chimici e chimico-fisici, dovuta probabilmente alle differenti cultivar di provenienza dei limoni, alle condizioni pedoclimatiche e alle tecniche di coltivazione. I risultati ottenuti per la frazione aromatica, mostrano come i componenti dell'aroma dei limoni di partenza vadano incontro a numerose trasformazioni, sia durante la preparazione del liquore che durante la conservazione, soprattutto catalizzate da un ambiente acido acquoso, dalle alte temperature e dalle radiazioni UV.

## INTRODUCTION

The world market of alcoholic beverages is changing. The demand of the consumers appears clearly directed toward medium alcoholic drinks and "natural" products. In this context, a large number of products have been introduced on the market. Among these, "limoncello", an Italian liqueur made with lemons, occupies a relevant part of the market. The success of this drink, greater than expected, is probably due to its freshness and moderate degree of alcohol as well as to its digestive qualities. Its popularity is even more astounding when compared to the overall crisis that liqueur market is experiencing (REDAELLI, 1997; BERETTA, 1996).

The traditional recipe for preparing a good limoncello calls for 1/2-1 kg of pesticide-free lemon peels, which are allowed to stay for one week to 40 days in one litre of 95% vol. alcohol. The mixture obtained is filtered and a syrup made with 0.8-1 L of water and 0.8-1 kg of sugar is added. The product thus obtained should contain about 30% sugar and 30% alcohol vol.; the remaining is water and the components extracted from the peels.

Limoncello, according to EC Regulation 1576/89 (O.J., 1989), can be defined as an alcoholic beverage made with fruit. The same EC Regulation prohibits the addition of natural flavour components originating from the same fruit used to make the liqueur, the addition of natural identical flavours and of colouring substances. Considering the strong market that limoncello has gained, it is necessary to establish some parameters of quality and authenticity.

The lemon tree (*Citrus limon* (L.) Burm. f.) in Italy is widespread along the Ionian and Tyrrenian coasts of Sicily and Calabria, in some limited areas of the Sorrento and Amalfi coasts and on the most protected banks of Lake Garda. The different varieties of lemon are distinguishable by the shape of the leaf and fruit, their productivity and the content of juice, seeds and essential oil, as well as for their resistance to temperature variations and diseases.

In general, the lemon blossoms several times during the year. The first blossoming gives winter lemons that mature between mid September of the same year and May of the following one. The second blossoming is typical of cultivars such as "Femminello comune", "Femminello siracusano", "Femminello S. Teresa" and "Femminello continella" which give "bianchetti" fruits, that mature from February to March. The third blossoming gives "verdelli", that mature from the end of April to September. Femminello is certainly the most common lemon cultivar in Italy, representing 70% of the total production. Along the Sorrento coast, "Ovale di Sorrento", that probably coincides with "Femminello comune", and "Sfusato Amalfitano" are the typical lemons. The first one is surely the best known and is traditionally used for the production of limoncello in that area (SPINA, 1985).

The literature reports numerous data on the composition of the volatile fraction of lemon essential oil, which represents about 96-98% of the whole oil. Limonene is the main component (60-70%), followed by β-pinene (9-17%) and γ-terpinene (7-11%) for the hydrocarbon fraction, and neral and geranial (citral) for the fraction of oxygenated compounds (COTRONEO *et al.*, 1986).

The quantitative composition of lemon varies according to the production period. Winter lemons, "bianchetti", and "verdelli" have characteristic differences (DUGO G., 1994).

Obviously, the organoleptic characteristics of the aromatic fraction of limoncello are very important in defining the quality of the product and are linked to problems of stability of the aroma in alcoholic beverages. In fact, citrus essential oils can undergo various oxidative alterations, isomerizations and hydrations because of the presence of unsaturated terpenes. Usually these reactions are influenced by temperature, light and pH (IWANAMI *et al.*, 1997; CLARK and CHAMBLEE, 1992; TEISSEIRE, 1994).

The literature does not report any scientific work on the composition of this product. Hence, this investigation was carried out to determine quality parameters for limoncello. In particular, the following were considered:

- The possibility of differentiating a genuine limoncello from an alcoholic drink obtained by adding natural lemon essential oil, or soluble lemon aroma, or distilled lemon oil to an alcoholic syrup, or also from a drink obtained adding reconstituted lemon oil and colouring substance to the syrup.
- The possibility of differentiating an artisan limoncello from an industrial product.
- The need to indicate an expiration date on the bottles.

## METHODS AND MATERIALS

The research was carried out on eight samples of limoncello produced in Calabria, two produced in Sicily, one from Sorrento provided directly by the producers and two found on the market. The following analyses were carried out on all the samples:

a) determination of the degree of alcohol (% vol.);

b) determination of pH with a potentiometer at 20°C;

c) determination of the total acidity expressed as citric acid, by titration with NaOH using a pH-meter as indicator;

d) determination of the percentage of sugar, as sucrose (RIZZATI and RIZZATI, 1998);

e) determination of total polyphenols as Folin-Ciocalteau index (RIZZATI and RIZZATI, 1998);

f) determination of ash and alkalinity of the ash;

g) determination of the aromatic fraction.

The determinations of chemical and physico-chemical parameters were carried out following the methodologies indicated in EC Regulation no. 2676/90 (O.J., 1990).

The components of the aromatic fraction were determined by gas chromatographic analysis of the fraction obtained by solvent extraction.

Each sample of limoncello (10 mL) was extracted with three 1 mL portions of n-hexane. The organic layers, collected and with the addition of an internal standard (n-dodecane, about 1 mg/mL), were injected in the gas chromatograph and analysed under the following conditions: Carlo Erba 5160 Mega Series gas chromatograph (Milan, Italy), equipped with a SE-52 fused silica capillary column 30 m x 0.32 mm i.d., film thickness 0.40-0.45 μm; a split/splitless injector and a flame ionisation detector (FID), both held at 250°C; injection mode: splitless; volume injected: 1 μL; carrier gas: He, 100 kPa (3.5 mL/min at 45°C); column temperature, 45°C (6 min) to 250°C at 3.0°C/min. Peak identification was carried out by GC/MS analysis using a Fisons GC8000 series gas chromatograph (Milan, Italy) equipped with a split/splitless injector, coupled to a MD-800 mass spectrometer. Data acquisition was performed with Mass-lab data acquisition. The GC was equipped with a Restek RTX-5MS, 30 m x 0.25 mm i.d., 0.25 mm film thickness; a split/splitless held at 250°C; injection mode: splitless; volume injected: 1 μL; carrier gas: He, 25 kPa (28.6 cm/sec at 50°C); column temperature, 50°C (6 min) to 240°C at 2.0°C/min, then to 280°C at 20°C/min. MS scan conditions: source temperature, 200°C; interface temperature, 250°C; E energy, 70 eV; mass scan range: 39-350 amu.

The yield, linearity of the method and correction factors were calculated using a mixture obtained by mixing some components (α-pinene, limonene, linalool, geranial, neral, geranyl acetate, β-caryophyllene) of the aromatic fraction at different concentrations with an alcoholic (30% vol) syrup.

## RESULTS AND CONCLUSIONS

Table 1 reports the chemical and physico-chemical composition of the limoncello samples. Each value is the average of three determinations.

Numerous studies (DI GIACOMO and CALVARANO, 1972; KUNKAR, 1990) on the composition of citrus fruit report that the peel of lemon fruit contains different organic acids, such as citric, malic, oxalic and mainly ascorbic acid. These acids are present as salts, so the pH of limoncello is almost always above 5.

Mineral components present in lemon are mainly cations belonging to the alkaline and alkaline-earth groups: potassium is the main component. The peels are rich in calcium, present both as pectate and oxalate. Magnesium is also present.

The composition of the ash is significantly influenced by several factors, such as the cultivar, pedoclimatic conditions and cultivation techniques. In fact, results differed greatly even for those samples produced in the same factory, under the same conditions but at different times (see samples 4 and 5).

Regarding the degree of alcohol, all the samples had values above the minimum allowable, that is 25% vol. Some samples from Calabria reached 37% vol., while the average value was 30% vol.

The percentage of sugar also varied greatly with values ranging from 18 to 29%. Among the samples analysed, sample 2 had the lowest alcohol content and a low sugar content, indicating that this sample contained a higher amount of water.

The content of phenolic substances, as indicated by the Folin-Ciocalteau index, varied greatly, due both to fruit (cultivar, origin, cultivation techniques) and method of production.

Table 1 - Chemical and physico-chemical parameters of limoncello samples.

| N. | Sample (origin) | Ingredients on the label | pH | Acidity (meq/L) | Alcohol (% vol.) | F.I. | Ash ‰ | Alkalinity of ash mg/L | Sugars (g/L) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | Reggio Calabria (Calabria) | Aqueous alcohol; Sugar; Lemon juice and infusion; Natural colouring material 28% vol. | 7.0 | - | 27.1 | 0.96 | 0.39 | 35.1 | 211 |
| 2 | Vibo Valentia (Calabria) | Hydroalcoholic infusion of lemon peels; sugar 25% vol. | 5.7 | 0.06 | 25.3 | 3.88 | 0.27 | 17.4 | 222 |
| 3 | Reggio Calabria (Calabria) | Alcohol; Sugar; Infusion of lemon peels; Natural aroma 32% vol. | 7.4 | - | 33.0 | 0.44 | 0.37 | 14.2 | 179 |
| 4 | Vibo Valentia (Calabria) | Water; Alcohol: Sugar; Infusion of lemon peels: Natural aroma 35% vol. | 5.6 | 0.09 | 36.9 | 2.48 | 0.40 | 2.4 | 286 |
| 5 | Vibo Valentia (Calabria) | Water; Alcohol: Sugar: Infusion of lemon peels: Natural aroma 35% vol. | 6.4 | 0.03 | 36.9 | 2.48 | 0.26 | 10.0 | 286 |
| 6 | Vibo Valentia (Calabria) | Aqueous alcohol; Sugar: Alcoholic infusion of lemon peels; Natural aroma 30% vol. | 6.0 | 0.03 | 31.4 | 1.60 | 0.18 | 6.0 | 277 |
| 7 | Cosenza (Calabria) | Water; Sugar: Alcoholic infusion of lemon peels; 32% vol. | 6.3 | 0.03 | 29.4 | 3.96 | 0.20 | 11.6 | 248 |
| 8 | Reggio Calabria (Calabria) | Water: Alcohol: Sugar: Infusion of lemon peels: 32% vol. | 5.5 | 0.15 | 33.5 | 16.24 | 0.53 | 15.8 | 277 |
| 9 | Market | Sugar: Infusion of fresh lemon peels: 30% vol. | 6.2 | 0.03 | 31.0 | 1.78 | 0.20 | 9.1 | 179 |
| 10 | Palermo (Sicily) | | 3.9 | 0.12 | 31.8 | 1.40 | 0.16 | 11.6 | 198 |
| 11 | Taormina (Sicily) | Sugar; Water: Alcohol: Fresh lemon peels: 28% vol. | 6.5 | 0.05 | 30.2 | 1.40 | 0.28 | 11.1 | 201 |
| 12 | Sorrento | Water; Sugar; Hydroalcoholic infusion of lemon peels; 30% vol. | 6.6 | 0.04 | 32.6 | 2.12 | 0.40 | 10.3 | 216 |
| 13 | Market | Sugar; Infusion of fresh lemon peels; Natural aroma; 30% vol. | 5.3 | 0.06 | 33.2 | 0.56 | 0.06 | 6.2 | 283 |

F.I.: Folin-Ciocalteau index.

Certainly, the most characteristic fraction of limoncello is the aromatic fraction, that comes from the alcoholic extraction of the essential oil contained in the peels. Table 2 reports the composition of the aromatic fraction as the relative % of the total aromatic fraction. The average values of the composition of the volatile fraction of lemon oil have also been reported (DI GIACOMO and MINCIONE, 1994). Fig. 1 shows a typical GC-FID chromatogram obtained for a sample of "limoncello" prepared in the laboratory following the traditional recipe using "verdello" lemons, and analysed immediately.

Extraction carried out on the laboratory mixture of standard components as described in the methods and materials section permitted the yield of the extraction method to be determined: it was more than 98% for all the compounds. The relative standard deviations (RSD%) of triplicate determinations of the standard solution were less than 5% for almost all the compounds.

As can be seen from Table 2 and Fig. 1, in comparison to lemon oil "limoncello" samples had lower amounts of mono- and sesquiterpene hydrocarbons and higher amounts of oxygenated components. This is in accord with the procedure used to make limoncello. In fact, hydrocarbons have a lower solubility in alcohol than oxygenated compounds.

During preparation and/or storage of limoncello, some hydrocarbons can undergo hydration reactions, mainly catalysed by acidic medium. Sample n. 10 had a very high content of $\alpha$-terpineol, together with a high content of terpinen-4-ol. Sabinene and $\beta$-pinene, which are co-eluted, had a value less than 1%. It is well known (CLARK and CHAMBLEE, 1992; VERZERA *et al.*, 1992) that $\beta$-pinene, as well as $\alpha$-pinene, under acidic aqueous conditions, give mainly $\alpha$-terpineol, while other monoterpene hydrocarbons, such as a-thujene and sabinene, give terpinen-4-ol as the main product.

From Table 1, it can be seen that sample n. 10 had the lowest pH (3.9), while the other samples were always higher than 5, ranging from 5.3 and 7.4.

The aqueous acid-catalysed reactions of terpenes do not require light, while other reactions, such as oxidation, often require light, usually UV light, to be initiated. These reactions are often associated with the formation of off-flavours.

Data from the literature report that citral (neral and geranial) is very sensitive to UV light, and may easily undergo photoreactions. Limonene, terpinolene, nonanal are also very sensitive to UV irradiation and their content decreases, while p-cymene increases during irradiation (IWANAMI *et al.*, 1997). Fig. 2 shows a GC chromatogram of a sample of limoncello (n. 12) where several unknown peaks absent in the essential oil of lemon were detected. These compounds, probably produced by degradation of aromatic compounds extracted from lemon peels, may be an indication of an old sample, or of a sample exposed to light (UV radiation) or high temperature.

The ageing of limoncello can be easily observed comparing values obtained for samples n. 4 and 5. In fact, these two samples were produced under the same conditions, by the same producer, from lemons of the same variety, but at different times. In more detail, sample n. 4 resulted older than sample n. 5: p-cymene, terpinen-4-ol, $\alpha$-terpineol were higher in n. 4, while limonene and citral were lower in sample n. 4.

After production, aromatic compounds in limoncello may undergo degradation, accelerated by light, high temperature and acid medium. For this reason it is necessary to report the expiration date on the bottles and also some indication as to the best storage conditions.

Samples n. 2 and 6 had very low values for all the monoterpene hydrocarbons. The main monoterpene hydrocarbons usually present in lemon oil (limo-

Table 2 - Composition of the aromatic fraction of limoncello samples (relative % of the total aromatic fraction).

| | Components | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | RSD% | Lemon oil |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | α-thujene | 0.3 | 0.2 | 0.4 | 0.2 | 0.3 | 0.2 | 0.4 | 0.3 | 0.4 | 0.1 | 0.3 | 0.2 | 0.3 | 7.2 | 0.4 |
| 2 | α-pinene | 0.8 | 0.4 | 0.5 | 1.3 | 1.0 | 0.3 | 1.5 | 1.3 | 0.7 | 0.6 | 0.7 | 1.0 | 0.7 | 5.5 | 2.0 |
| 3 | camphene | 0.1 | ND | ND | 0.1 | 0.1 | ND | 0.1 | 0.1 | ND | 0.1 | ND | ND | ND | - | 0.1 |
| 4 | sabinene+ β-pinene | 11.2 | 3.4 | 4.6 | 9.7 | 10.5 | 3.3 | 12.2 | 10.7 | 7.6 | 0.7 | 5.2 | 15.5 | 5.2 | 4.83 | 14.8 |
| 5 | 6-methyl-5-hepten-2-one | ND | ND | 1.5 | 0.3 | 0.2 | ND | ND | ND | ND | ND | ND | 0.3 | ND | - | ND |
| 6 | myrcene | 1.6 | 0.6 | 2.2 | 1.3 | 1.6 | 1.9 | 1.0 | 1.4 | 1.3 | 0.7 | 1.1 | 0.6 | 1.1 | 3.7 | 1.5 |
| 7 | α-phellandrene + octanal | 0.3 | 0.3 | 0.3 | 0.3 | 0.5 | 0.8 | 1.1 | 0.2 | 0.1 | 0.1 | 0.2 | 0.7 | 0.2 | 7.9 | 0.1 |
| 8 | α-terpinene | ND | ND | ND | 0.2 | 0.2 | ND | 0.2 | 0.2 | ND | ND | ND | ND | ND | - | 0.2 |
| 9 | *p*-cymene | 2.4 | 1.0 | 2.0 | 2.6 | 1.5 | 1.6 | 2.4 | 0.4 | 1.1 | 5.6 | 1.5 | 6.6 | 1.5 | 4.6 | 0.2 |
| 10 | limonene | 56.0 | 13.5 | 39.0 | 46.8 | 53.1 | 22.4 | 50.1 | 47.6 | 36.2 | 40.5 | 42.5 | 34.9 | 42.5 | 4.6 | 65.4 |
| 11 | (Z)- β-ocimene | ND | ND | ND | 0.1 | 0.1 | ND | 0.1 | 0.1 | ND | ND | 0.1 | ND | 0.1 | - | 0.1 |
| 12 | (E)- β-ocimene | ND | ND | 0.2 | 0.1 | 0.2 | 0.3 | 0.1 | 0.2 | 0.2 | ND | 0.1 | 0.1 | 0.1 | 13.5 | 0.1 |
| 13 | γ-terpinene | 2.4 | 1.8 | 2.3 | 4.7 | 5.4 | 3.0 | 7.8 | 7.1 | 6.5 | 3.4 | 4.5 | 3.6 | 4.5 | 4.5 | 9.5 |
| 14 | cis-sabinene hydrate | ND | 0.3 | 0.4 | 0.1 | 0.5 | ND | 0.3 | 0.2 | 0.9 | ND | 0.4 | 1.4 | 0.4 | 4.1 | 0.1 |
| 15 | octanol | ND | 0.5 | ND | 0.3 | 0.1 | 0.6 | 0.2 | 0.2 | 0.2 | 0.3 | ND | 0.5 | ND | 6.4 | ND |
| 16 | terpinolene | 0.3 | 0.3 | 0.8 | 0.3 | 0.3 | 1.7 | 0.3 | 0.4 | 0.5 | 0.7 | 0.3 | 0.2 | 0.3 | 10.8 | 0.4 |
| 17 | *trans*-sabinene hydrate | ND | 0.8 | 0.2 | 0.2 | 0.4 | 0.2 | 0.4 | 0.4 | 0.4 | ND | 0.1 | 1.1 | 0.1 | 4.3 | ND |
| 18 | linalool | 1.6 | 3.6 | 1.5 | 1.5 | 1.2 | 4.5 | 1.0 | 1.6 | 1.2 | 0.7 | 1.1 | 1.7 | 1.1 | 7.1 | 0.1 |
| 19 | nonanal | 0.2 | 0.8 | 0.3 | 0.6 | 0.6 | 1.0 | 0.3 | 0.4 | 0.4 | 0.3 | ND | 1.5 | ND | 6.0 | 0.1 |
| 20 | citronellal | ND | ND | 0.4 | 0.1 | 0.3 | ND | 0.1 | ND | 0.3 | 0.5 | ND | 0.5 | ND | 8.5 | 0.1 |
| 21 | borneol | ND | ND | ND | ND | ND | ND | ND | 0.1 | ND | ND | ND | ND | ND | - | ND |
| 22 | terpinen-4-ol | 0.8 | 12.0 | 1.2 | 4.0 | 1.0 | 5.6 | 4.4 | 2.2 | 1.2 | 6.5 | 3.9 | 2.1 | 3.9 | 3.7 | ND |
| 23 | α-terpineol | 0.8 | 8.8 | 1.1 | 3.9 | 2.6 | 6.5 | 2.6 | 2.3 | 1.4 | 23.8 | 4.0 | 4.4 | 4.0 | 3.5 | 0.2 |
| 24 | decanal | 0.2 | 0.3 | 0.5 | 0.3 | 0.2 | 0.6 | 0.2 | 0.2 | 0.3 | 0.2 | 0.4 | 0.6 | 0.4 | 4.7 | 0.1 |
| 25 | nerol | 0.6 | 14.4 | 2.6 | 1.5 | 1.6 | 4.0 | 2.1 | 4.1 | 8.6 | 2.8 | 1.1 | 5.0 | 1.1 | 3.7 | 0.1 |
| 26 | neral | 7.2 | 5.0 | 15.0 | 3.5 | 4.8 | 10.7 | 2.5 | 2.9 | 7.2 | 0.5 | 10.0 | 1.9 | 10.0 | 4.1 | 0.9 |
| 27 | geraniol | 0.6 | 16.5 | 1.4 | 1.9 | 1.7 | 3.3 | 2.5 | 4.6 | 4.0 | 4.9 | 1.1 | 3.2 | 1.1 | 4.6 | ND |
| 28 | geranial | 8.1 | 5.8 | 18.0 | 4.5 | 6.1 | 12.9 | 2.9 | 3.7 | 8.4 | 0.5 | 11.8 | 2.4 | 11.8 | 3.9 | 1.5 |
| 29 | citronellyl acetate | ND | 0.7 | ND | 0.3 | 0.1 | 0.3 | 0.1 | 0.6 | 0.4 | 0.3 | 0.3 | 0.3 | 0.3 | 3.0 | ND |
| 30 | neryl acetate | 2.0 | 3.5 | 1.8 | 3.5 | 1.6 | 5.4 | 1.2 | 2.9 | 4.2 | 2.3 | 4.3 | 5.9 | 4.3 | 3.7 | 0.4 |
| 31 | geranyl acetate | 2.5 | 3.3 | 1.5 | 4.7 | 1.5 | 6.6 | 1.3 | 2.2 | 4.1 | 2.9 | 3.7 | 2.7 | 3.7 | 3.0 | 0.4 |
| 32 | β-caryophyllene | ND | 1.2 | 0.5 | 0.5 | 0.3 | 0.5 | 0.2 | 0.3 | 0.5 | 0.2 | 0.4 | 0.2 | 0.4 | 2.8 | 0.2 |
| 33 | *trans*-α-bergamotene | ND | 0.2 | ND | 0.3 | 0.1 | 0.4 | 0.2 | 0.5 | 0.2 | 0.2 | 0.3 | ND | 0.3 | - | 0.4 |
| 34 | β-bisabolene | ND | 0.4 | ND | 0.4 | 0.2 | 0.5 | 0.2 | 0.4 | 0.5 | 0.4 | 0.4 | 0.2 | 0.4 | - | 0.5 |
| 35 | 2.3-dimethyl-3-(4-methyl-3-pentenyl) 2-norbornanol | ND | 0.2 | ND | 0.2 | 0.1 | 0.4 | 0.1 | 0.1 | ND | ND | 0.2 | 0.2 | 0.2 | - | ND |
| 36 | campherenol | ND | 0.3 | ND | 0.2 | 0.1 | 0.3 | 0.1 | 0.2 | 0.4 | 0.2 | 0.2 | 0.4 | 0.2 | - | ND |
| 37 | α-bisabolol | ND | 0.2 | ND | 0.2 | 0.1 | 0.4 | 0.1 | 0.1 | 0.3 | 0.1 | 0.2 | 0.5 | 0.2 | - | ND |
| 38 | nootkatone | ND | ND | ND | ND | 0.1 | ND | ND | ND | 0.4 | ND | ND | ND | ND | - | ND |

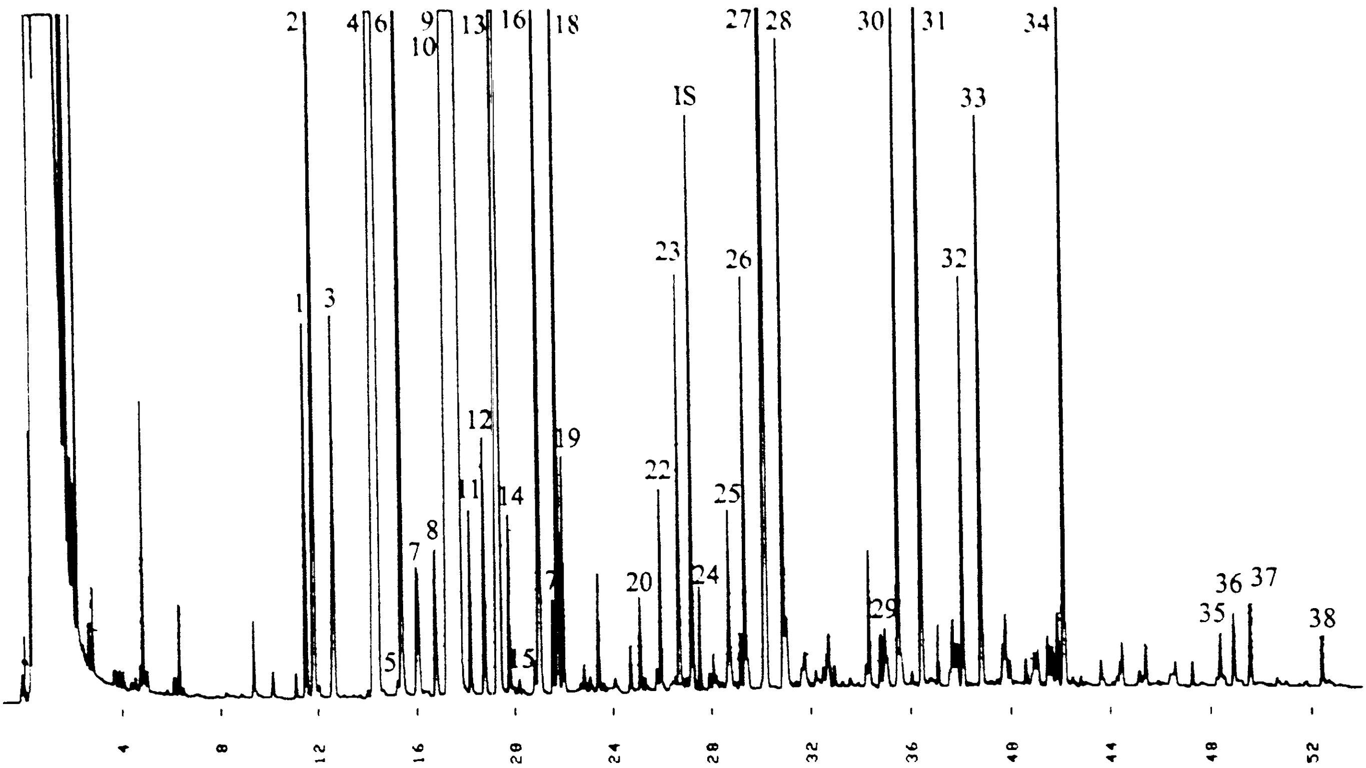


Fig. 1 - Gas chromatogram of the volatile fraction of a limoncello sample prepared in the laboratory. For peak identification. see Table 2. IS: internal standard.

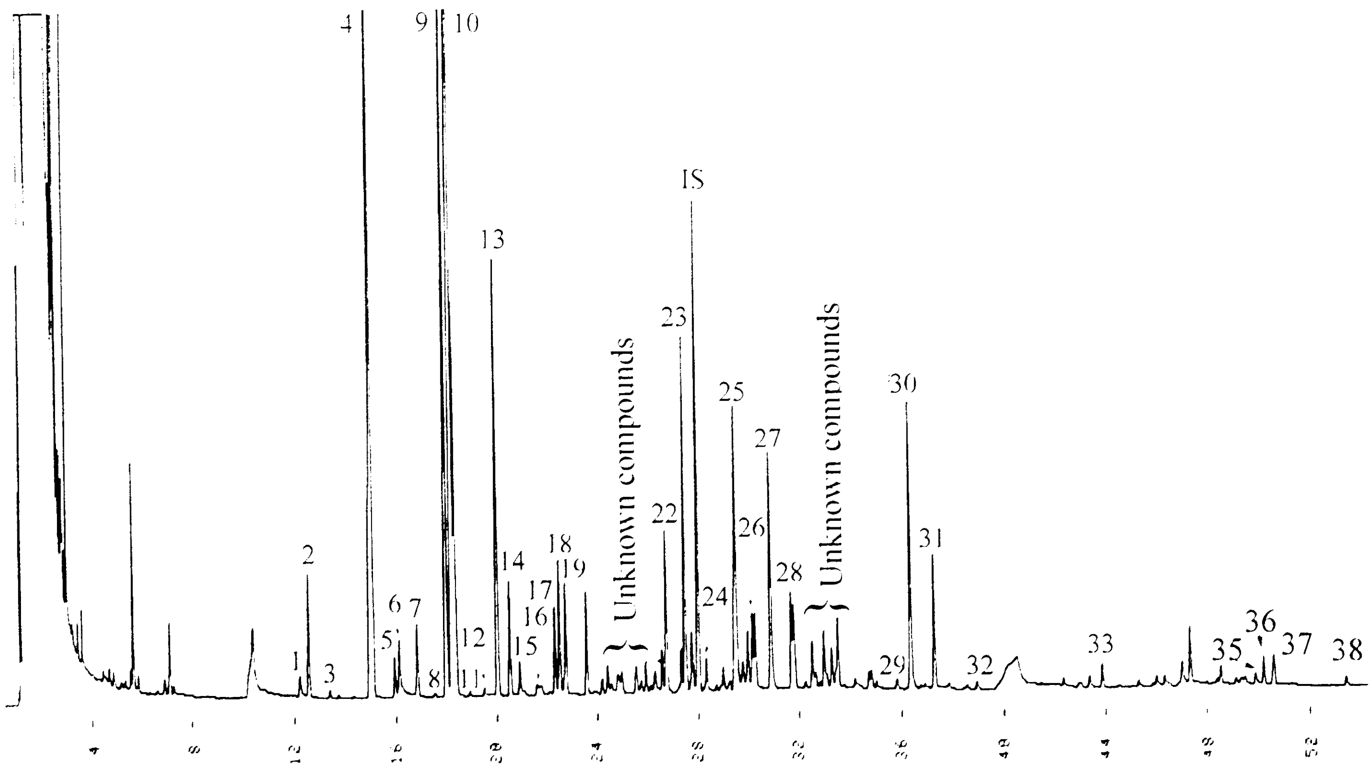


Fig. 2 - Gas chromatogram of the volatile fraction of limoncello sample n. 12. For peak identification, see Table 2. IS: internal standard.

nene, β-pinene, γ-terpinene) were very low and the % of oxygenated compounds was quite high. Comparing these values with those of a deterpenated lemon oil, it may be possible that these samples of limoncello were made by adding a deterpenated oil and a colouring material to the alcoholic syrup. This procedure allows the preparation time of the product to be shortened and production costs to be reduced.

In conclusion, data reported in this paper show that limoncello liqueurs present on the market have a different quantitative composition. In particular, during the preparation and storage, the conditions of low pH, high temperature and light, that significantly modify the composition of limoncello aroma should be avoided.

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Revised paper received December 22, 1999 Accepted February 23, 2000