Use of Sr-resin for cheese geographical origin classification

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Outline:

- Introduction: the pecorino cheese from Farindola
- $^{87}Sr/^{86}Sr$ ratio as a tracer for geographical origin
- Isotopic composition analysis by Termal Ionization Mass Spectrometry (TIMS)
- Problems of interferences
- Use of Sr-resins for Sr separation
- Preliminary results

A unique and tipical product from Farindola village:

Farindola village

Pecorino di Farindola

Consortium for the protection of the original “pecorino di Farindola” against food frauds

Purpose of our work:

Investigate potentiality and limits of using precise $^{87}Sr/^{86}Sr$ ratio measurements to discriminate pecorino cheeses of different geographical origins

Variable isotopic composition of strontium

^{84}Sr ^{86}Sr ^{87}Sr ^{88}Sr

$$^{87}Rb \rightarrow {}^{87}Sr + \beta^- + \overline{\nu}_e$$

Naturally occurring β-decay of ^{87}Rb

$T_{1/2} = 4.7 * 10^{10}$ y

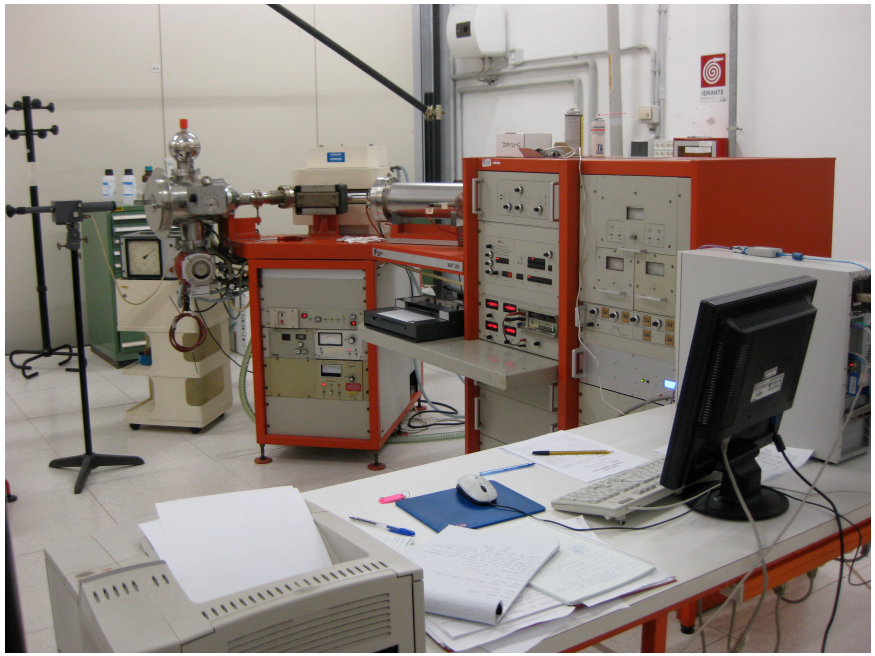
- **$^{87}Sr/^{86}Sr$** ratio reflects variations in the radiogenic ^{87}Sr in soils
- strontium isotopic composition of a sample can give information about its **geographical origin**
- biological processes (animal methabolism) do not significantly fractionate strontium isotopes
- measured Sr content in pecorino cheese: ≈10 ppm (ICP-MS)

- *G. Fortunato et al, JAAS (2004) 19, 227-234*
- *P.R. Trincherini et al, Food Chemistry 145 (2014) 349-355*

Highly precise isotopic analysis:

Thermal Ionization Mass Spectrometry (TIMS)

- Discrimination between isotopic ratio values: <0.1%
- Internal precision of the measurement: >0.005%



Why Sr-resin from Triskem®?

Elements affecting the measurement

^{87}Rb – ^{87}Sr isobaric interference → Mandatory Sr – Rb separation!

Ca | Na | K

Content of the mainly interesting elements in pecorino matrix (ICP MS semi-quantitative measurement)

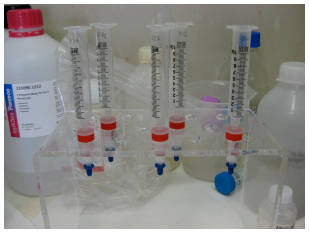
Element	[ppm]
Na	≈ $8*10^3$
K	≈ $1*10^3$
Ca	≈ $3*10^3$
Rb	≈ 1
Sr	≈ 10

Sample mineralization:

1. ≈ 1.5g of homogenized sample in a quartz crucible; thermal decomposition @ 700°C for 3 hours

2. Dissolution in 10mL of 4M HNO_3 (1 hour @ 95°C)

3. Sr-resin



4. Dry evaporation

5. Dissolution in 25 μL of 1% ultra-pure HNO_3 solution

6. Deposition on the renium filament of the TIMS source
   (≈ 1-5 μg of analyte)

Sr-resin:

1. rinsing (5 mL of 8M HCl)
2. sample loading
3. 5mL of 2M HNO3 (fraction #1)
4. 5mL of 8M HNO3 (fraction #2)
5. 5mL of ultra-pure water (fraction #3, elution of Sr)

Element	Solid cheese [ppm]	Fraction #1 [ppm]	Fraction #2 [ppm]	Fraction #3 [ppm]
Na	$7.6*10^3$	$1.3*10^3$	1.1	<0.01
K	$0.9*10^3$	$0.2*10^3$	$0.1*10^3$	<0.1
Ca	$3.6*10^3$	$0.6*10^3$	1.0	1.0
Rb	**1.0**	0.2	0.001	**<0.0007**
Sr	**5.3**	<0.003	<0.003	**4.8**

(The concentrations are reported to the solid cheese sample)

Notes:

1. Excellent Rb-Sr separation!

2. Good Sr recovery (90%)

3. It is proved that the Sr resin **does not** cause srontium isotopic fractionation

G. Fortunato et al, JAAS (2004) 19, 227-234

suitable for our purpose

Preliminary results:

10 samples of pecorino from Farindola producers

Sample	#65	#67	#62	#21
$^{87}Sr/^{86}Sr$	0.70932	0.70946	0.70932	0.70933

Sample	#27	#74	#22	#37
$^{87}Sr/^{86}Sr$	0.70933	0.70893	0.70912	0.70941

	Isotopic standard NIST SRM 987
$^{87}Sr/^{86}Sr$	0.71034

Next steps:

1. Analyze batches of equivalent samples from other regions (of Italy and of the world)

2. Data statistical analysis and data comparison

3. Integrate this study with data from other analysis (mineral composition...)

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Consortium for the protection of “pecorino di Farindola”


...and thank you all!