



Textile Tracker

Isotope analysis for the proof of origin of textiles.

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accredited according to
DIN EN ISO/IEC 17025:2018

Background

Check of origin with stable isotopes

Water plays an essential role in stable isotope traceability. The mean isotopic ratios of hydrogen and oxygen in precipitation are primarily dependent on the annual temperature of specific locations [Dansgaard 1964] and secondarily on other less influential factors such as altitude, latitude and the continental effect [Araguas 2000]. Consequently, there is great variability in isotopic patterns in groundwater geographically [Bowen 2002]. Hydrogen: $\delta^2\text{H}$ and oxygen: $\delta^{18}\text{O}$ isotopes in plant cellulose reflect the isotopic patterns observed in groundwater with some modifications, the signature is primarily influenced by the evaporation effects of water [Flanagan 1991a] and secondarily by biochemical fractionation in the anabolism of cellulose [Sternberg 1986, Luo 1992].

However, there is still a linkage to the water pattern existing in the cellulose.

The information is present at the elementary level in the hydrogen and oxygen of the cellulose and can hardly be changed.

This is the reason why the stable isotope method is considered as the universal method for verifying the origin. For example, the European Union refers to the stable isotope method in its guidelines for the verification of wood (also a cellulose product).

Therefore, the stable isotopic method is applicable to cotton as well and can even be used in textile. That was already verified from Agroisolab in a project funded by DBU.

Besides that, the cellulose in cotton is offering further stable isotopic information which is reflecting the origin. So, there is a strong fractionation in carbon ratios: $\delta^{13}\text{C}$ that is dependent on stomata conductance and photosynthetic assimilation [Farquahar 1982]; both are influenced by environmental factors such as humidity, light and temperature. Therefore, the carbon ratio in cellulose reflects the local climate of the area in which it grew and is suitable as an additional parameter to add resolution for tracking the origin using stable isotopes.

Finally, the stable isotopic information of nitrogen $\delta^{15}\text{N}$ and sulfur $\delta^{34}\text{S}$ are referring to fertilization and geological situation of the soil.

This information is still contained in the cotton as residual amounts of peptides / amino acids.

The fertilization parameter: $\delta^{15}\text{N}$ is regularly used to check organic farming [Bateman 2007]. The $\delta^{34}\text{S}$ signature is normally used to verify the local origin of the product [Thode 1991]. However, big patterns as high depleted ratios in Canada or enriched patterns in Latin-America are applicable for origin checks as well.

It should be mentioned that Agroisolab is certainly one of the few stable isotope laboratories in the world that uses accredited methods even in cotton.

Thus, the analytical results of Agroisolab are also used in legal cases [Camin 2017]

Influence of processing

Within the current state of knowledge, the typical processing steps as washing, boiling, bleaching and mercerization don't have any relevant influence on the stable isotopic signatures of carbon, hydrogen, oxygen and nitrogen. Which could be successfully demonstrated in a DBU funded project (37085-01: 2021 to 2022).

However, dyeing with nitrogen-active compounds (e.g. Azo-) always has negative effects on the isotopic ratios of nitrogen. Also, the isotopic signatures of sulfur are only well usable in unprocessed cotton.

Nevertheless, many more complex production steps, such as hydrophobic treatment, easy-care finish, pigment print can be eliminated by adapted extraction in the laboratory. These protocols have been successfully developed by Agroisolab in the mentioned DBU project.

Summary:

- *The isotopic patterns of hydrogen, oxygen, and carbon are usually robust in processed cotton especially when extraction protocols are considered.*
- *Nitrogen and sulfur can only be used if no interference is expected (e.g. coloring).*

Mixing of cotton with technical fibers.

Without any doubt Agroisolab prefers to analyze 100 % cotton in textiles. Nevertheless, often an analysis of mixtures is required.

This is possible if the additional component can be separated beforehand. Currently, a protocol for polyester has been developed and the possibility of separating up to 50 % polyester has been validated.

If the polyester exceeds this validation limit, it does not mean that the protocol cannot be used, but the protocol is used outside the validated range. Further protocols for the separation of technical fibers e.g. spandex are under development.

However, it should be mentioned that the separation of viscose is not possible because both cotton and viscose ultimately consist of cellulose.

A stable isotope analysis then only makes sense if the proportion does not exceed 3 %, because as a rule the induced error due to these foreign substances lies within the variation range of the measurement.

Summary:

- *100 % cotton in textiles is preferred*
- *Separation protocols are available for polyester (max: 50 %).*
- *Without separation, the foreign content must not exceed 3%.*

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Current countries in the database

Agroisolab is constantly working to expand the database for origin determination.

The aim is to build up a global database with partners, as has already been achieved in the WFID initiative in wood with partners such as FSC. Currently the following countries are in the database:

- Australia
- Brazil
- Burkina Faso
- China-Shangdong
- China-Xinjiang
- Egypt
- India
- Ivory Coast
- Kazakhstan
- Kirgistan
- Mali
- Pakistan
- Peru
- Sudan
- Tansania
- Tschad
- Turkey
- Uganda
- USA
- Uzbekistan

In addition to open database, Agroisolab also builds custom database, which is used to monitor the supply chain of customers.

A special alternative of this is our IsoTrace concept, where supplier references are archived and compared in a "tier to tier" test if necessary. This concept has long been established in agricultural sector and follows the logic of an "anti-doping database".

Transportation to the laboratory

Since Agroisolab analyzes the elemental signature of the samples, there are no major specifications of the sample delivery. Only a minimum quantity of 50g (cotton) should be observed.

The indication of meta data like percentage composition (mixed samples), possible origins, or predominant origin are important information for our work.

A short information of sample shipment to the laboratory is always helpful, so that the Agroisolab team is already prepared and the rehearsal intake can proceed quickly. Of course, the customer is always informed about the sample receipt as well as the analysis runtime.

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