CULTIVATION AND EXTRACTION OF NATURAL DYES FOR INDUSTRIAL USE IN NATURAL TEXTILES PRODUCTION

Fact Sheet

Project Information

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Coordinated by
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Objective

The objectives of the project are the re-establishment of the cultivation of dye plants in the agriculture and their use for dyeing textiles in industry. In addition the project aims to develop ecologically sound and economically sustainable cultivation systems

- efficient low-cost dye recovery methods
- environmentally friendly textile dyeing
- cleaning of waste water
- decomposing of plant residues by natural processes.

The proposers suggest that to achieve this objective, modern economically
sustainable cultivation methods will have to be developed for important natural dye plants. In addition, efficient low cost extraction methods for dye stuffs must be found and the dyeing process must be optimised, taking into consideration aspects of human health and safety, as well as the environment. Based on present knowledge and historical reports, it would appear that the best dye plants for industrial use under European conditions are Rubia tinctoria (madder) for the colour red, Isatis tinctoria (woad) for the colour blue, Reseda luteola (weld) and Solidago sp. (golden rod) for the colour yellow. These species have been investigated in terms of cultivation practice and dyeing ability.

Up to now natural dyes have been neglected as renewable resources for agriculture although there is an increasing demand for environmentally friendly textiles produced from natural fibres (wool, silk, cotton, linen etc.) and natural dyes. The aim of the project is to carry out research to find out how natural dyes, cultivated in Europe, can be produced and used with economically sustainable and efficient processes. Modern cultivation systems will be developed for the most important natural dyes, woad (Isatis tinctoria), madder (Rubia tinctorum) weld (Reseda luteola) and golden rod (Solidago ssp.) which deliver the rare and important colours blue, red and yellow.

Experimental trials carried out from the first partner in England and from the second partner in Germany will find out the best conditions for the growing of dye plants in regard to soil and climatic factors. The two partners will develop modern cultivation systems for getting maximal dye yields including optimal seeding and harvesting time, optimal fertilisation of plants, efficient weed control and safe preservation procedures. Plant material harvested from field trial will require preparation for use in dyeing. The utilizable parts of the plants will have to be subject to specific dehydration processes or have the dyestuff extracted, or possibly both, to make them usable in dyeing and possible to store. The third partner, a dye-house in Germany, will be using the plant extracts made by the project partners. The essential parameters for the dyeing procedures have to be examined. The addition of the dyes and of dyeing auxillaries will have to be optimized and to be adapted for the different fibres according to the required technical and environmental standards. An environmental dyeing process requires also an adequate treatment and for the utilization of all residual material. The aim is to establish an effective recycling system for all remainders.

Cropping:
So far, reliable methods of cultivation, including seeding, fertilisation and weed control, have been developed for Rubia tinctoria and Solidago. In addition, the best harvesting time and storage techniques for harvested material, as well as methods of seed extraction, have been established for these crops. For the other two (madder and weld), similar investigation continue. Difficulties have been found in the cultivation of these two latter crops. The seed set of madder shows significant variation from year to year under middle European conditions. In some cases no seed is formed. So far this has been overcome by planting runners. A solution of this
problem may be to produce the seed in the more southern parts of the EU. The problems with golden rod (Solidago canadenis) is a result of the very small seeds, which produce seedlings with poor survival rates. So far, the best method of propagation appears to be seeding in a greenhouse, followed by planting out of young plants in the field. Since golden rod is perennial the additional costs for this procedure are not so significant.

Storage:
The dye in the dried material of weld, golden rod and madder seems to be relatively stable during storage in the dark. Before dyeing, the dye can be extracted with boiling water. Immediate processing of the fresh harvested plant material may give a higher yield of dye. Woad differs in that indigo itself does not exist. There are two precursors of indigo in Isatis: indican and isatan B (which is very unstable). Drying of blades of Isatis leads to a significant loss of dye pigments. Alternative methods of indigo preservation such as freezing or freeze drying did not yield promising results. It was found difficult to remove the indigo from leaves adequately by any simple means. Therefore, the best method would appear to be the extraction of precursors followed by indigo formation.

Indigo formation:
The precursors of indigo can be extracted from woad at any temperature - very quickly in boiling water (minutes) and slowly at low temperature (2 to 3 days at 20 °C). A prolonged extraction reduces the yield. The best extraction temperature seems to be 40 50 °C and a steeping duration of one day. The indigo content of the precipitate is only 10 20 %. However, this is not a drawback. This raw material gives more interesting different shades of a clear blue colour in comparison to indigo from Indigofera tinctoria. The fastness to rubbing for Isatis is better than that for indigo from Indigofera. However, indigo from Indigofera can be bought from India at a very low price of around ECU 20 per kg. Hence, at present, the low content of precursors in the present cultivated woad in addition to competition prevents the use of Isatis as an indigo plant. But this disadvantage could be overcome by breeding varieties with higher mass yield and higher content of indigo precursors.

Dyeing:
Parameters for the dyeing process (such as dye stuff concentration, time of extraction of the raw material, influence of temperature, mordant concentration and best pH) were determined for Reseda, Solidago and Rubia. The extracts were used for dyeing wool, silk and cotton under optimised conditions. The results from one type of textile could be transferred easily to other natural textiles. The same colour will develop, only the intensity may differ. Textiles made with fibres of animal origin (wool, silk) generally gave a higher intensity than ones of plant origin (cotton, linen). Excellent clear colours were obtained, even though for environmental reasons only mordant solutions without heavy metals were used. Quality control of the dyed textiles was carried out according to German and International Standards for
fastness to washing, washing at 37 °C, to saliva and perspiration, to water and in terms of resistance to rubbing. The results obtained indicated a high quality according to the standards.

Wastes and effluents:
Experiments concerning the composting of the remainder material from dye plant extraction, waste water treatment and microbiological tests were started. It was found that ultrafiltration gave a clear waste water fraction and a concentrated dye fraction which will be tested for additional possibilities of dyeing procedures.

FURTHER WORK

In addition to the four main species, more potential dye plants were cultivated and investigated in regard to their mass yield and dyeing abilities. The following aspects were noted: growth, mass yield, possibilities of mechanical seeding (drilling) and harvesting of seeds. Some of them have a high yield potential. Preliminary dyeing experiments suggest there are species which may have higher dye contents and/or greater dye strength than Reseda or Solidago and with a shade similar to the latter. These include Serratula tinctoria, Chrysanthemum vulgare, Centaurea jacea and Anthemis tinctoria.

CONCLUSIONS

The dye producing plants investigated are mainly wild types. Nevertheless, many of them can be adapted to modern cultivation practices for industrial use at low costs and at a high yield of useful products. The trials of the different dyes on various textiles were so promising that it is anticipated that the use of such dyes would occur on an industrial basis in the near future.

Many plants produce stable colorations or pigments which have been used as dyes in the past. Their use decreased as organic dyes based on coal and other fossil feedstock became available at lower cost. This project is looking at the feasibility of reversing this trend and providing high quality natural dyes from plants, creating new opportunities for both farmers (as growers) and the fabric industry - in line with the current consumer trends towards natural products. The group has investigated four possibilities in detail, madder for red and woad for blue, as well as weld and golden rod for yellow. In addition, a wide range of other species have been looked at. Most gave good dyeing performance when judged by German and internationally-accepted standard tests. However, problems remain to be solved in production of planting material, yield and (in the case of indigo) competition against low cost imports. The work continues under the original AIR funding.

ACTIVITIES

Cultivation.
Cultivation methods have been developed and tested for growth of woad (Isatis tinctoria), madder (Rubia tinctorum), weld (Reseda luteola), Canadian golden rod (Solidago canadensis), dyer camomile (Anthemis tinctoria), safflower (Carthamus tinctorium) and ai (Polygonum tinctorium). Cultivation studies included investigations of the best seeding time, seeding density, row distance, fertilising, weed control, optimal harvest time, harvest methods, preservation procedures to get maximal biomass and especially maximal dye yields. Questions of seed production and optimal seed multiplication were also investigated. The results obtained so far have been used to write recommendations concerning cultivation methods.

Harvest and Storage.
The dye stuffs in the dried material of weld, golden rod, madder, dyer' camomile, safflower and ai are relatively stable during storage in a dry dark room, as was shown by comparative dyeing experiments using fresh and one year stored plant material. Generally the dyeing power depends to a significant extent on the time of harvest and the drying conditions.

Dyeing.
Dyeing experiments can be done with aqueous extracts, preserved with food preservatives. Such extracts are suitable for use in small scale applications. For the larger quantities, used for industrial purposes, aqueous extracts are not acceptable due to the weight in transport. Dyeing experiments with whole plant material using powered material, which could be contained in a cloth bag, gave good results. These methods are suitable for dyeing on the small scales. The bag dyeing method is convenient and a simple method for DIY and arts and crafts. For industrial use the problems of handling and transport must be solved by making a powder.

Processing.
Storage in a dried state is unsuitable for the plants producing a blue dye. The indigo formed in the leaves by drying represents only a small part of the total indigo precursors. It is also difficult to remove the indigo from the leaves by simple means. Therefore aspects of precursor extraction and subsequent indigo formation have been investigated. The extraction of indigo from Polygonum tinctorium simpler and the yield is very much higher than that for woad. The raw material contains a maximum of 20 % of indigotin in the case of woad, but about 40 % in Polygonum. Dyeing tests with indigo samples with different indigotin content have shown that the quality of dyeing depends on the content of dye stuff.

Complexing agents.
In order to optimise the dyeing procedure, the influence of complexing agents was investigated. Studies included investigation of the effect of pH, the ratio between bottom mordant/textile weight and the advantages or disadvantages of the dyeing in a two-bath or a single-bath process.
Optimisation.
The dyeing process needs to be optimized to enhance the fastnesses to washing, to perspiration, to light and to resistance to rubbing, in order to increase acceptance of natural dyes by the textile industry. Special attention needs to be paid in order to optimise the dyeing processes for the many different techniques used in dindustry, such as loose dyeing, rope dyeing, dip dyeing and mechanical dyeing.

Waste treatment.
Attempts to develop an ecological acceptable method for waste water treatment were very successful. Good results for the purification of waste water were obtained with activated carbon, although all other materials investigated were found to be ineffective. The flow rate of waste water, operating convenience, reprocessing of the absorbent and low costs are essential parameters for choosing the best filtration method for activated carbon. For smaller quantities of waste water (DIY and art and crafts) it is convenient to work in batches. For larger quantities and industrial use activated carbon was tested in continuous working systems. A cricket filter enabled optimal flow conditions. High degrees of purity of the filtrate and high flow rates are combined with short regeneration times and filtration cycles. Chamber filter presses are recommended for smaller quantities of waste water and for processes working in batch technique. Deep bed filtration in only economical for small quantities. The advantage is a very clean and easy method. The most elegant and economic method proved to be reverse membrane diffusion. The method allows an effective and easy filtration with high flow rates.

Programme(s)

Topic(s)

Funding Scheme

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