

ADVANCED GCE
APPLIED SCIENCE
Sampling, Testing and Processing

G628

INSERT

Monday 17 January 2011
Afternoon

Duration: 1 hour 30 minutes



INFORMATION FOR CANDIDATES

- Questions 1 and 2 are based on the articles which follow on pages 2–5 of this Insert.
- This document consists of **8** pages. Any blank pages are indicated.

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Woad – a plant of antiquity with modern uses

The deep blue dye indigo has been used in Europe since Neolithic times. Until the 16th century the only source of indigo was from the woad plant (*Isatis tinctoria*), Fig. 1a, which originated in Western Asia but has been cultivated throughout Europe since ancient times. Some books record that the Ancient Britons painted their bodies with woad when fighting the Roman invaders. However, there is some doubt about this as it is ineffective as a body paint and is too astringent for use as a tattoo.

In York there is evidence that the Vikings used woad as a source of indigo. Medieval uses of indigo were not limited to textiles and some early Christian book illustrations use a woad-based pigment as a source of blue paint.

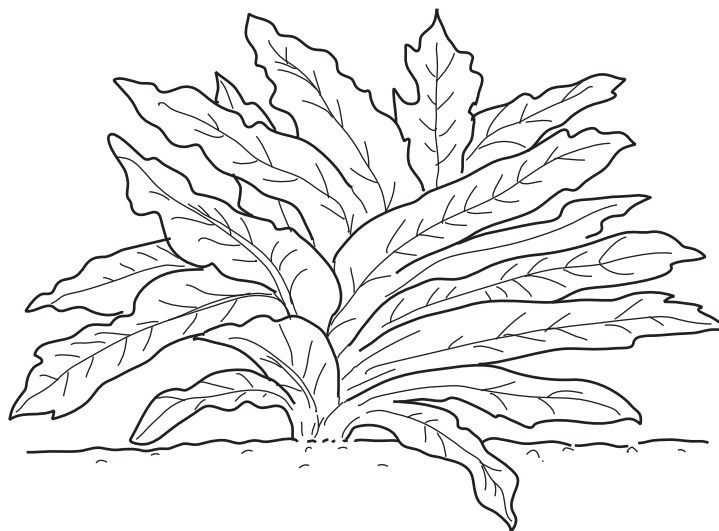


Fig. 1a

From the 17th century the use of woad, in Europe, as a source of indigo declined. The dye was increasingly obtained from the tropical shrub, *Indigofera tinctoria*, as foreign trade increased. In Japan, indigo was obtained from the *Polygonum* plant.

In the last century the production of indigo from plants was almost entirely replaced by synthetic indigo made from naphthalene, a product of coal tar. At present the production of indigo by this method, discovered in 1897, is about 17 000 tonnes per year.

However, the traditional production of indigo from plants has not entirely died out. In 2004 the European Union sponsored a project to produce natural indigo from woad on a commercial basis. Extensive research identified the most suitable varieties of woad, growing conditions and methods of extraction for optimum indigo production. In Britain the Government was interested in the manufacture of inkjet dyes from plants, including woad. Products derived from woad are biodegradable unlike current inkjet dyes.

Woad is a biennial plant but its leaves are harvested for dye production during the first year. The production of indigo from woad is a labour intensive process. In outline, the picked leaves are washed and then treated with hot water to extract soluble materials. After cooling, solid material is removed and the solution remaining is made alkaline, producing a green-brown solution. The mixture is then aerated, when indigo slowly separates as a blue solid. Traditionally the leaves were allowed to ferment in the presence of urine but this stage has been replaced by a substitute!

Unfortunately indigo cannot be used for direct dyeing as it is not soluble in water. Instead, indigo is reduced to a compound that gives a green-brown solution and the prepared fabric is then soaked in a vat containing this solution. On removal of the fabric from this solution and subsequent exposure to air, oxidation to indigo occurs giving a blue-dyed fabric. Indigo is still much in demand as a blue dye for dyeing denim jeans. It has the disadvantage that it slowly fades but that is part of the attraction of clothes such as jeans.

There is increasing interest in naturally occurring materials that may help to combat cancer. Woad is a member of the *Brassica* family, which also includes cabbage and broccoli. There is some evidence that glucobrassicin (GBS), present in *Brassic*as, has anti-cancer properties. GBS is produced by woad as a natural insecticide and is present in much higher quantities in this plant compared to broccoli. GBS contains nitrogen and sulfur and researchers fed the woad plants with nitrogen and sulfur containing fertilisers in the hope of increasing the yield of GBS. They also bruised the plants to simulate insect damage to see if the plants would produce more GBS. The results were encouraging because the amount of GBS contained within the plant was four times the amount of GBS present in those plants acting as a control. The mass of GBS present in these treated plants was around 1% of the mass of the dried plant. This encouraging percentage will enable researchers to obtain enough GBS so that they can study further the metabolism and anti-cancer properties of this material.

The search for phosphorus – an essential element

In 2005, the residents of Bassingbourn, in Cambridgeshire, voted on a proposal to erect a statue on their village green. The statue was to consist of four large bronze balls, each representing coprolite – fossilised dung.

This village was one of the centres of coprolite mining in the nineteenth century. Coprolite is the scientific term for a phosphatic nodule that is largely composed of calcium phosphate together with some marine animal remains.



Fig. 2a

Coprolites have often been thought simply to be dinosaur droppings but this is only partly true as many contain remains of other animals including sea dwellers. It is thought that many of these creatures died out about 65 million years ago because of a rise in sea level and tidal waves caused by an asteroid impact. These fossilised remains, particularly coprolites, form a layer up to two metres thick across a large stretch of East Anglia.

Farmers found that ground-up coprolite material enabled crops to grow better. This was because of their high phosphate content, which is very much higher than that from other sources, such as guano – largely seabird excrement. Coprolites were mined from pits, dug up to six metres below ground level. Generally the coprolite miner was working in a layer up to one metre in thickness. The peak period for coprolite mining was 1850 to 1885. After this time the most accessible seams had been worked out and large, more easily worked layers of phosphate-containing rock had been discovered abroad. Today, little is to be seen of this, once thriving, industry. The only evidence is shallow depressions in fields.

The United States has considerable reserves of shales that contain phosphatic nodules, presumably of organic origin. One large-scale example of these shales is the Pleasanton shales that are found in Kansas. Considerable research has been carried out to find an easy and economic method to extract and concentrate the phosphate rich rock from the shale. If this could be achieved, this material would provide a considerable additional source of phosphorus.

At present however, the principal phosphate ore is apatite – this is commonly known as ‘phosphate rock’. Apatite is a complex mineral that contains mainly calcium phosphate, carbonate and fluoride. Most of this rock is mined by open-cast and quarrying methods.

Much of this ‘phosphate rock’ is converted to the fertiliser ‘superphosphate’ by reaction with sulfuric acid. More soluble fertilisers such as diammonium hydrogenphosphate are also manufactured. Some of the phosphate rock is converted into various sodium phosphates. These have extensive uses in the detergent industry and there is also some need for these compounds in the food industry.

The analysis of phosphate-containing rocks is not easy. There are two basic problems. The first is the separation of the phosphate from the many other compounds in the rock that would interfere with the method being used and prevent accurate results being obtained. The other is the lack of a quick and straightforward method of quantitative analysis for phosphate. Traditional weighing methods (gravimetric analysis) involving ammonium magnesium phosphate can be used, for example, or colorimetry using 'molybdenum blue', where the phosphate reacts with a molybdenum compound to give a deep blue colour in solution. However, both of these methods are time consuming and can present difficult problems in procedure.

We have come a long way since 1851, when the miller, John Ball, first ground coprolites to a powder and used it to fertilise his soil to grow better turnips. The population of the world has increased enormously and the search for phosphate rocks from which the mineral can be extracted easily and economically still presents a considerable challenge.

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