

## **RESULTS TASK 2**

### **EXTRACTION OF THE OIL AND TANNIN FROM THE SEEDS BY WORKING FIRST ON A LABORATORY SCALE**

#### **OIL**

##### **Grease content and other soluble substances in solvents.**

To be able to perform the tannin extraction in an aqueous medium, the grape waste oil content must be previously extracted. Firstly the oil was extracted by a Soxhlet system with dichloromethane and later with hexane which is less toxic.

Size particle has resulted to be of importance to the oil content extraction result. The smaller size the better the oil extraction ration. So, on the basis of particles of a size of 150-250 $\mu$  or even less than 150 $\mu$ , values exceeding 20% of oil content can be obtained using hexane.

The qualitative characterisation of oil extracted with dichloromethane from crushed seed (size particle over 400  $\mu$ m) by TLC, Thin Layer Chromatography, showed the following greases: mineral oils, cerids, methylic esters, triglicerides and fatty acids. By means of Chromatography of Gases we know that 34% of fatty acid compounds are saturated compounds and 56% of them are unsaturated compounds (21% are monounsaturated compounds and 35% are polyunsaturated which are convenient for human health).

Distilleries send seed to oil producing companies for oil extraction and commercialisation to pharmacies and the food industry.

#### **TANNINS**

##### **Tannin extraction**

Tannins are insoluble with cold or hot water.

To extract them it is necessary the treatment of crushed seed with metabisulphite and temperature, in aqueous medium, in autoclave in order to introduce sulphonic groups and increase their solubility.

The autoclave allows us to work at higher temperatures than 100°C and reach upper atmospheric pressure. The extraction process was optimised in order to increase the tannin content in solution.

##### **Characterisation**

In order to test the tannin's properties we assessed the new tanning extract solution in comparison with solutions of extracts of chestnut, quebracho, mimosa, gambier and other tannins from crushed vegetable such as tara, sumac, bark of mimosa, takaout and granadine.

Test of coagulation was used to know the kind of tannin (condensed or catechin tannins // hydrolysable or pyrogallic tannins). Grape tannin from seed is a condensed tannin but it is partially hydrolysable as well as chestnut.

On chromatography thin layer grape tannin from seed has a behaviour similar than quebracho (catechin).

The precipitation with metallic ions (Al, Co, Cu, Fe and Zn), hydrogen peroxide, and calcium hydroxide was tested. In general, grape tannin's results using metallic ions were similar as chestnut, except for the amount of precipitate produced using copper and iron. It proves its semi-catechin nature.

The precipitation with surfactant proved that any tannin precipitated with dodecylsulfonate (anionic surfactant) whereas all tannin precipitated with nonilfenoletoxilate and lightly cationic surfactant (in this case because of the anionic properties of tannins). It was again proved similar results between chestnut and grape seed.

Grape tannin from seed has bacteriostatic properties like the current vegetable tannins.