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Analysis of various physical properties of oleoresin obtained from *Pinus patula* in Amarkantak, Madhya Pradesh, India

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Abstract
Oleoresin are among the most important NTFPs in the World. They are one of the oldest tree products used by humans. The *Pinus patula* tree planted globally as a dominant plantation forestry crop across various parts of the world especially in the high altitude tropical environment of India. Analysis of various physical properties were carried out in the laboratory conditions. The findings revealed that the oleoresin samples exhibited a number of properties that could be useful to the chemical and pharmaceutical sectors, but no commercial oleoresin tapping experiments have indeed been conducted in this species to far. The resin samples were found to be highly soluble in Chloroform and having high rosin content of (91.48%).

Keywords: Oleoresin, *Pinus patula*, Tropical pines, Tupentine oil, Viscosity

1. Introduction
Resin is mostly a mixture of volatile monoterpenes and non-volatile diterpenic resin acids. Through a distillation process, resin can be separated into turpentine (30%) and rosin (70%) (Hawley and Palmer 1912) [7]. *Pinus patula* Schlecht. &Cham commonly known as Mexican weeping pine or Jelecote pine native to Mexico (from Tamaulipas to Oaxaca region).This species has been widely planted for plantation forestry program on a large scale in Africa, Columbia, Brazil, Argentina, India, Nepal, New Zealand (Farjon, 2017) [6]. It grows fast and produces a long, straight bole, for most part free of branches and the wood obtained from this tree is soft, light colored and easily worked, therefore it finds application in flooring and paneling, plywood and other wood based industries. In India first trial plantation of *Pinus patula* was raised in West Bengal in 1929 at Ramam which was located at an elevation of 2270 m, Whereas in the present study site it was planted at Different compartments of Amarkantak range during 1970 at an elevation of 1080 m amsl. This oleoresins obtained from Pinus tree act as one of the most important non-wood forest products As per records, more than 80 species of pine trees are tapped for the production of resin throughout the world (Hillis,1987). The purpose of this study was to determine physical properties of *Pinus patula* oleoresin samples**.** This properties affects the processing and makes this nature of studies necessary so as to determine the predictability of the end product and consequently quality of the output.

2. Materials and Methods
Trees selected for oleoresin tapping were located in Amarkantak Forest Range,Madhya Pradesh at an elevation 0f 1060 m amsl and lies at 22 ^{0} 41’ N latitude and 81 ^{0} 43’ longitude. These areas were selected for provenance trail of many tropical pine plantations in early 1970s under a Co-ordinate research program of Forest Research Institute.For oleoresin tapping, three distinct methods were used to collect samples, that were then examined in the laboratory for various parameter estimations.

2.1 Determination of Turpentine and Rosin content
Twenty-five grams of oleoresin sample from each tree was taken· in a round bottom flask and sufficient amount of water was added to it. The flask was fixed with Clevenger's apparatus and was heated with frequent agitation. Heating was continued for about one and half hour and after cooling for at least five minutes; the volume of turpentine in the graduated portion of the tube was noted. The distillation was continued until successive readings of the volume of turpentine did not differ (Persad, 1983) [13].

Calculation of per cent Rosin & turpentine content

$$\text{Turpentine (\%)} = \frac{X}{\text{Sample weight (25gm)}} \times 100$$

Where: x is the volume of turpentine in ml.

2.2 Rosin content

The non-volatile solid material called rosin which remained in the flask after distillation was allowed to cool until it was solidified. The rosin was weighed and the percentage of rosin was calculated as under:

$$\text{Rosin(\%)} = \frac{X}{\text{Sample weight(25gm)}} \times 100$$

Where: X is the weight of rosin in gram.

2.3 Determination of solubility

Solubility is defined as the amount of substance that passes into solution to achieve a saturated solution at constant temperature and pressure. Solubility is expressed in terms of maximum volume or mass of the solute that dissolve in a given volume or mass of a solvent. The solubility of the resin was determined in, acetone, chloroform and benzene. 1.0 g sample of the resin was added to 50 ml of each of the above-mentioned solvents and left overnight. 25 ml of the clear supernatants were taken in small pre-weighted evaporating dishes and heated to dryness over a digital thermostatic water bath. The weights of the residue with reference to the volume of the solutions were determined using a digital top loading balance (Model.XP-3000) and expressed as the percentage solubility of the resins in the solvents (Mohsenin,1978).

$$\text{Water solubility index (\%)} = \frac{\text{Weight of dissolve resin sample in solution(g)}}{\text{Weight of resin sample(g)}}$$

2.4 Determination of Moisture content

Moisture content of resin samples was determined by drying 5g of the resin sample to constant weight at 80 ^{0}C using hot air oven. Dried samples were cooled in desiccators before weighing. Moisture content was expressed as percentage of mass loss from the original mass as described by Yusuf (2011) [18].

$$\frac{\text{Weight of total sample} - \text{Weight of dry sample}}{\text{Weight of total sample}} \times 100$$

2.5 Determination of Ash content

Ash content of the resin samples was determined by burning 1g of resin sample in a muffle furnace at 550 ° C for 4 hours. The ash content was expressed as % ratio of the weight of ash to weight of the sample.

Ash value is designed to measure the total amount of material remaining after ignition. It includes bothphysiological (derived from plant tissue itself) and non-physiological (residue of the extraneous matter like sand that are adhering to plant substances. It also used to determine the critical levels of foreign matter, acid insoluble matter, salts of calcium, potassium and magnesium (Mocak *et al.* 1998).

$$\text{Ashcontent(\%)} = \frac{W3 - W1}{W2 - W1} \times 100$$

Where

W1 = weight of the empty crucible,

W2 = weight of crucible + sample and

W3 =weight of crucible + ash

2.6 Determination of Viscosity

The viscosity of a liquid is its resistance to shearing, to stirring in capillary tube. Viscosity was considered as one of the most important analytical and commercial parameters, since it is a factor involving the size and the shape of the macro-molecule. The viscosity was determined and calculated for the 4%, concentration of all the resin sample at room temperature and rotational speed of 100 rpm by using spindle 62 and Spindle 63 of digital Brookfield DV-E viscometer (Sinha, 2017) [16].

2.7 Determination of Colour and Odour

The collected resin sample was visually analysed for colour identification and aroma authentication through smells.

3. Results and Discussions

3.1 Turpentine and Rosin content

Both components were separated from the resin using steam distillation using Clevengers apparatus. The two major products obtained from pine tree resin are turpentine oil and rosin. In the present experiment it was found that turpentine oil which is volatile in nature was found tobe 8.52% and rosin content was 91.48% as shown in figure 2.Rosin or colophony is the solid fraction and has numerous uses, such as paints, varnishes, coatings, adhesives and other applications related to polymers (Maiti *et al.* 1989; Arrieta *et al.* 2017; Narayanan *et al.* 2017) [10, 2, 12]. Whereas the turpentine oil fins a wide variety of industrial uses such as in perfume industry, disinfectants and synthetic pine oil etc. The colour of rosin samples were found to be light yellow in colour. Coppen and Hone (1995) [5] reported that Rosin is usually translucent with a color ranging from light yellow to brown. Among different diameter class selected for resin tapping diameter classes of 35-40 cm yielded better resin yield. Teshome (2011) [17], observed that diameter class of 25-30 cm produced highest turpentine oil contain.

3.2 Solubility

The resin samples of *Pinus patula* was found to be highly soluble in Chloroform (1.28%), followed by Acetone and Benzene shown in figure 1.Schafer and Zandbiglari (2002) [15] revealed that Epoxy resin was highly soluble in Chloroform than Eucalyptus oil. The current finding also found satisfactory as compared to the report made by above workers.

3.3 Moisture content

The moisture content of resin samples was determined using hot air oven. It was observed that the moisture content of resin sample was 1.1%. Jalali *et al.* (2011) [9] also reported that the moisture content in oleo gum resin of *Ferula gummosa* was 6.7%.Whereas moisture content in resin samples of *Pinus roxburghii* collected from Kashmir Valley was 3% (Ahmad *et al.* 2011) [1]. These figures were far higher than those observed in the current experiment.

3.4 Ash content

The ash content in the resin was determined by muffle furnace. The values of Ash content in Rosin samples and Resin samples were found to be 21% & 1% respectively. The higher value of ash in rosin samples was because of due to the

volatile component turpentine was separated by distillation method. Whereas in resin samples turpentine oil was there. Saeidy *et al.* (2018) [14] reported in the oleo gum resin of Asafoetida (*Ferula assafoetida)* having ash content of 2.75%.The resin of *Pinus patula* had a 1% ash content in the current experiment; this difference could be attributed to the chemical characteristics of the two species being different.

3.5 Viscosity

The viscosity of different resin samples was calculated by using Brookfield viscometer at a rotation of 100 rpm with the use of spindle (No. 62 and 63) and at room temperature (approximately 32°C).From the laboratory analysis it was found that the viscosity value by using 62 and 63 number needles are 0.90 cp and 1.20 cp (Centipoise) respectively. Bourdeau and Schopmeyer1958 reported that rate of oleoresin flow seems to be determined by Viscosity, number and size of resin ducts.

3.6 Colour and odour

Pinus patula having strong camphor like aroma with creamy white and yellowish white color respectively. Cabaret *et al.* (2019) [4] conducted Spectro-colorimetry study in maritime pine (*Pinus pinsater*) and revealed that two different colour observed in this species. In the present experiment similar colour variation was observed. This colour variation illustrated a possible difference in the chemical composition.

Table 1: Physical properties of *Pinus patula* resin samples

Physical properties	Value
1. Turpentine oil (%)	8.52
2. Rosin (%)	91.48
3. Solubility (%)	
a. Acetone	1.07%
b. Benzene	0.92%
c. Chloroform	1.28%
4. Moisture content (%)	1.1
5. Ash content in resin (%)	1
6. Ash content in rosin (%)	21
7. Viscosity	
a. 62 number needle	0.90 cp
b. 63 number needle	1.20 cp
8. Colour	Yellowish white and Creamy white
9. Aroma	Strong camphor

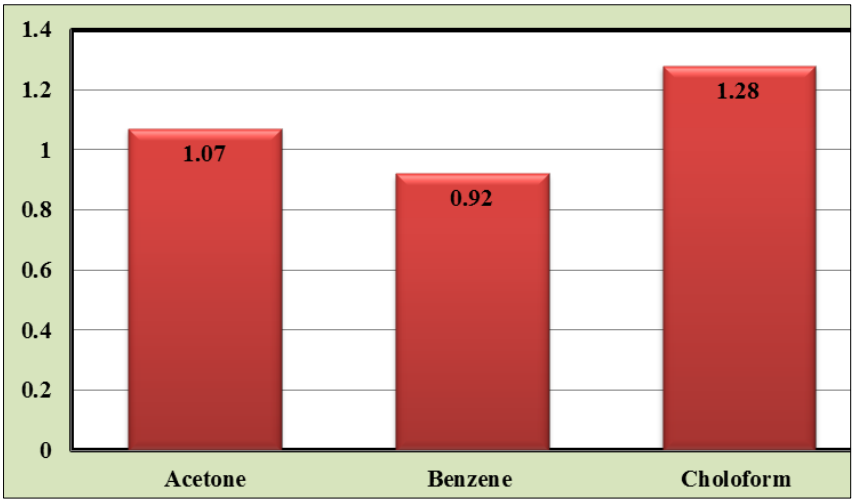

Fig 1: Solubility of resin samples in different organic solvent

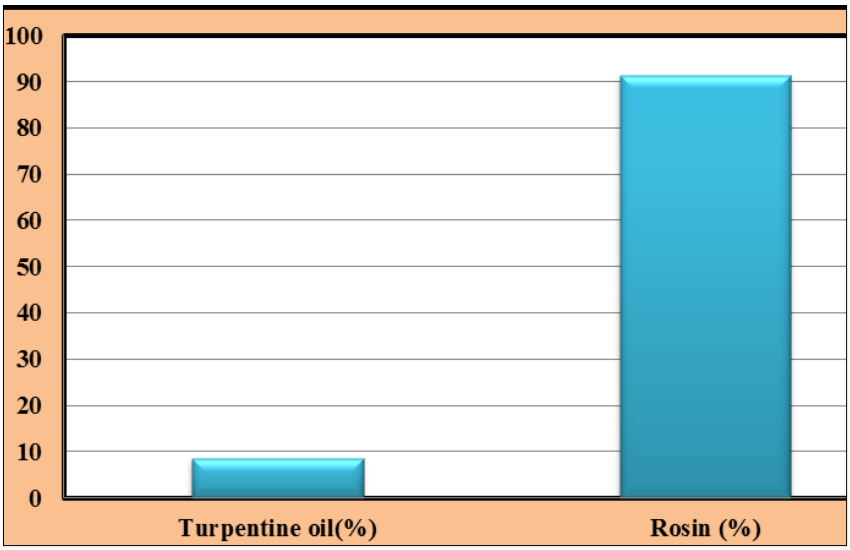

Fig 2: Rosin and Turpentine oil content *in Pinus patula* resin samples

Fig 3: Preparation for solubility test

Fig 4: Turpentine oils after distillation Exudation of oleoresin from *Pinus patula* tree

Fig 5: Estimation of Viscosity of resin Samples by using Brookfield DV-E Viscometer

Fig 6: Estimation of Ash content of resin samples in Muffle furnace

Fig 7: Separation of Rosin and Turpentine oil by *Clevenger apparatus* (L), Ash content in Rosin samples

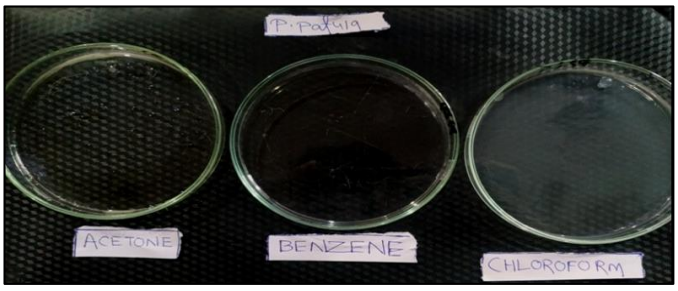

Fig 8: Solubility test in different organic solvent (Acetone, Benzene, Chloroform)

4. Conclusions

Physical properties of the pine oleoresin are vary from species to species this is because of due to genetic parameters. This species is categorized as Least Concern by the IUCN Red List, and its global population is robust. As a result, a sustainable method of resin tapping should be developed so that additional bio-physical investigations in this species can be conducted on a global scale.

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