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Original Article

Extraction and characterization of coconut (*Cocos nucifera L.*) coir dust

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Abstract

Studies were carried out on the characterization and extraction of coconut coir dust using water, acetone, acetone/ water (70/30), (50/50) respectively. The acetone extract of the coir dust was phytochemically screened for tannins, polyphenols, flavanoids, phlobatannins. The moisture, ash, lignin, and cellulose contents of the coir dust were determined by measuring the cation exchange capacity (CEC) and the pH were found to be 2.39 mmol g^{-1} and 6.4 respectively. The degree of swelling of coir dust increased with increased solvent contact time. The percentage extract showed the best solvent system to be acetone/water (70/30). In all the solvent systems, the smaller the particle size of coir dust, the greater the amount of extract. The phytochemical screening of the acetone extract indicated significant amount of tannins, flavanoids and other polyphenols in coir dust.

Keywords: characterization, coir dust, extraction, agricultural byproduct, phytochemical screening

1. Introduction

Coir dust is the light, fluffy material that falls off from thick mesocarp of coconut (*cocoos nucifera L.*) fruit when it is shredded during coir processing. The fibre is used for the manufacturing of ropes, carpets, mats, and related products. The fall off materials remain available as waste product of no industrial value and are normally incinerated or dumped without control (Vidhana and Somasiri, 1997).

However in advanced countries, coir dust is compressed into bricks or bales wrapped and shipped for sale (Evans *et al.*, 1996). They are used for preparing soilless growing media for cultivation of some ornamental plants or crops (Reynolds, 1974; Meerow, 1994). Some of the properties and compositions of this lignocellulosic material has been documented in the literature (Tejano, 1985; Joseph and Sarma, 1997; Abad *et al.*, 2002). Its constituents include cellulose, hemicelluloses, and extractives such as pectins and tannins. These constituents make coir dust a useful adsorbent or natural ion exchanger because of the hydroxyl and carboxyl groups present in its composition. Many lignocellulosic materials, mostly agricultural byproducts, have been utilized as biosorbents/ion exchangers for removal of cations/anions from solutions. Maize cob, sugarcane bagasse, peanut skin, banana pith, orange peel (Randall *et al.*, 1994; Low *et al.*, 1995; Igwe and Abia, 2003; Krishnani *et al.*, 2004; Perez-Marin, 2007). Of all the constituents of lignocellulosic material, the soluble components can be extracted with polar and non-polar solvents or combination of both. These extractives have widespread applications.

A typical example of soluble extractive from lignocellulosic materials, such as coir dust, is tannin, a polyhydroxy compound. It is a generic name given to a complex

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substance of numerous polyphenolic components with phenolic moities condensed into a complicated polymeric structure. Tannins are a very important wood product obtained from tree barks, stem, vegetables, and other plant parts. They are used in the food industry (wine making), medicine, adhesives, particle board, and leather based industries (Aaron, 1982). Wattle plant tannins from South Africa have been largely exploited and utilized in the production of tannin-based adhesives in leather and particle board based industries (Pizzi, 1978; Pizzi and Scharfetter, 1978; Pizzi and Roux, 1978).

Pansera *et al.* (2004) used four solvents comprising of acetone, methanol, ethanol, and water to extract tannins from acacia bark at 60°C. Hence, there is a need to investigate the extraction of soluble components in coir dust to determine factors that lead to better yield. An in–depth study of the characteristic of this promising adsorbent/ion exchanger and extraction of the soluble components would improve existing knowledge regarding the material. This will suggest the various ways it can be utilized; hence, coir dust, which was under-utilized can now be channeled to industrial uses. The objective of the study is therefore to characterize and extract coir dust of varying particle sizes using acetone, water and their mixtures.

2. Materials and Methods

Coir dust was procured from a local coconut processing mill in Uyo, Nigeria. It was air-dried for 24 hours and in an oven at 60°C to constant weight. The dried coir dust was separated into different particle sizes ranging from 63–800 µm, and stored in air-tight lid containers.

2.1 Extraction of coir dust

The solvent systems used were water, acetone, acetone/water (70/30), (50/50), respectively. The modified procedure of Pansera *et al.* (2004) in extraction of tannins from *Acacia mearnsii* was adopted.

Extraction in water: Coir dust (1 g) was added to 100 ml of deionised water in a beaker and stirred for 60 minutes at 100°C, the boiling point of the solvent. At the end of 60 minutes, the mixture was filtered and the filtrate was evaporated to dryness before the weight of the extract was obtained. The above procedure was repeated for different volumes of water ranging from (100–400 ml) for the various particle sizes (63–800 µm). The weight of extracts and residues were determined in all the experiments.

2.2 Extraction with acetone and acetone/water mixtures

Coir dust (1 g) was placed in the thimble set in the soxhlet extraction fitted into a round bottomed flask with solvent (80 ml) while 20 ml was added to soak the sample in the thimble. The whole set up was placed in a water bath at constant temperature of 60°C. The solvent was heated to reflux and the extraction completed in 60 minutes. The filtrate was distilled to remove the solvent and the extract dried to constant weight at 60°C. The same procedure was used for the different solvent systems, acetone/water (70/30) and (50/50) for each particle size.

2.3 Characterization of coir dust

The coir dust was characterized for moisture, ash, lignin, cellulose contents, pH, cation exchange capacity, degree of swelling and solubility in some solvents according to standard methods. Moisture content, cation exchange capacity (CEC) were determined using standard method of Tanigami *et al.* (2007). Ash content was determined according to the standard method of Association of Official Analytical Chemist (AOAC, 1975), while degree of swelling and solubility in solvents were determined using the procedure of Vasudevan and Sarma (1979) on composite cation exchangers. The presence of lignin and cellulose in the coir dust was determined using the phloroglucinol solution and Hierberman-Storch method (Browning, 1990), respectively.

2.4 Phytochemical screening of coir extract

Thirty milligrams (30 mg) of the acetone coir extract was dissolved in 20 ml of deionized water at room temperature with stirring and the resulting solution used for the phytochemical screening (Roux, 1951; Sofowora, 1984; Trease and Evans, 1989).

3. Results and Discussion

The coir dust was characterized by standard methods (AOAC, 1975; Browning 1990; Abad *et al.*, 2002) and the results are shown in Table 1. The moisture, cellulose, lignin, ash contents, water and dilute acid (HCl) extractives were 25.2%, 35.99%, 53.5%, 9%, 28.2%, and 41.3%, respectively. The solubility in aqueous NaOH solutions (1%) and (18%) were 27.5% and 41%, repsectively, while solubility in cold and hot water were 25% and 31%. The pH of the coir filtrate and cation exchange capacity were 6.4 and 2.39 mmolg^{-1}, respectively. The high lignin and cellulose with low ash contents of coir dust are characteristics of agricultural by-products generally referred to as lignocellulosic materials. These findings are in accordance with earlier studies on coir dust (Tejano *et al.*, 1979; Festin and Jose, 1985; Abad *et al.*, 2002; Carrijo *et al.*, 2002). These authors reported cellulose to vary between 23–43%, lignin 35–54%, ash 6-8.25%, while the rest were extractive components. These values, however, depend on the age, source, period extracted, and extraction method (Tejano *et al.*, 1985; Abad *et al.*, 2002).

The lignin and cellulose contents of coir dust are higher than those of other agricultural byproducts, such as corncob, rice straw, sugarcane bagasse, and cotton stalk. The cellulose content of rice straw is 37.2%, bagasse 46.2%, cotton stalk 42.8%, while the lignin content is 12.2%, 20.2%, and 27.3% respectively (Nada *et al.*, 2002). The high lignin and cellulose content of coir dust contribute to its physical stability, which does not change markedly with moisture content. Piles of coir dust have been found to last as long as 100 years without breaking down at coir processing centres (Meerow, 1994; Evans *et al.*, 1996). Lignin and cellulose are biopolymers bearing multiple phenolic hydroxyl, carboxylic, amino, sulphate groups admittedly associated to be responsible for the removal of heavy metals ions and other contaminants from waste water/effluents (Tan *et al.*, 1993; Veglio and Beolchini, 1997; Gballah *et al.*, 1997).

The low water extractives compared to that of diluted acid (HCl) may be attributed to the fact that in water the components of the coir dust are only hydrosolubilized, while both hydrosolubilization and hydrolysis of the coir constituents such as hydroxyl groups occur in acids (Pansera *et al.*, 2004). Hence the quantity of extractives from diluted hydrochloric acid is greater than from water.

Results of the alkaline solubility of coir dust in dilute NaOH solution (Table 1) showed that coir dust had the higher solubility in 18% NaOH solution. Its high solubility in dilute NaOH is due to the partial dissolution of waxes and resin extractives and also partial degradation of bonds between lignin and cellulose (Nada *et al.*, 2002). This increases with increase in concentration of the alkali solution; which leads to increase in number of components of the coir dust being released into the solution. It has been reported that non-woody agricultural by-products exhibit high solubility in dilute alkalis (Umoren *et al.*, 2004).

Ash content of coir dust is 9.0%, which is consistent with reported values of 6.0–8.3% (Tejano *et al.*, 1985; Festin and Jose, 1989). The low ash content of coir dust suggests that it contains low extractives with little or no waxes and resin when compared with other non-woody lignocellulosics materials, such as plantain stem (*Musa paradisiaca*) of 55.80%.

The solubility in cold water, 25%, and in hot water, 31%, gives an indication of the level of extractives and water soluble constituents of coconut coir dust. The higher solubility of coir dust in hot water may be attributed to decomposition of these components at high temperature. The extractives, waxes and resins, are the undesirable components of pulp and paper products of lignocellulosic material; hence their removal improves the quality of coir as pulp and paper raw materials (Casey, 1980). Another disadvantage of these extractives is that the rendering lignocellulosic materials are less effective as ion exchange/adsorbent matrix. They can mask the cellulose, lignin and hemicellulose, which bear most of the reactive sites in any lignocellulosic material. This will hinder the penetration of metal ions or other contaminants to the sites, hence, low sorption. Most agricultural byproducts are poor adsorbents/ion exchangers in the unmodified forms (Anirudhan and Unnithan, 2007).

The degree of swelling in water (Table 1) is in the range of 65-85% for 24 hours and depends on the contact time with the solvent. The degree of swelling of saw dust-based ion exchangers show similar trends in the range of 100-200% (Vasudevan and Sarma, 1979). Degree of swelling of adsorbents/resins is an important requirement for materials intended to be used as ion exchangers (Tanigami *et al.*, 2007).

Abad *et al.* (2002) reported a pH range of 5.2–6.3 and an exchange capacity of 3.17–6.98 mmol g^{-1} for coir dust samples from thirteen different sources. From Table 1, an average pH value of 6.4 and an exchange capacity of 2.39 mmol g^{-1} were obtained from the coir dust sample. The magnitude of the cation exchange capacity of a material is a measure of the quantity of its exchangeable ions. This depends on the number of active groups per unit weight of the resin. The higher the number of its exchangeable ions, the greater the value of exchange capacity. The above properties of coir dust together with its high porosity and capillary nature suggest its potential as a useful material for adsorption/ion exchange reactions.

3.1 Extraction of coir dust

The results of the extraction using water as solvent are shown in Table 2. The amount of residue obtained ranges from 53.2% to 97.1%, which is higher than the extracts (4.30% -27.0%). Coir dust contains high solid matter of over 90% of cellulose with lignin, while the extractives constitute less than 10% of its composition. Moreover, these constituents are cemented together through hydrogen bonds with crystalline regions. Hence, solubilization of this structure with water is hindered by these cementing materials (Sjostrom, 1993).

The amount of extract obtained from 1 g of coir dust for all particle sizes increased with the volume of water used for the extraction (Table 2). This increase in volume of water

Table 1. Characterization of coir dust.

Assay	% content in coir dust
Moisture Content	25.5
Ash Content	9.0
Water Extractive	28.2
Acid (Dil. HCl) Extractive	41.3
Swelling in H_2O	
1 hour	65.7
5 hours	70.8
10 hours	77.7
24 hours	85.7
Swelling in Acetone	0.934
Swelling in dil. HCl	3.229
pH of Coir Dust in H_2O	6.4
Cellulose	35.99
Lignin	53.5
Alkali Soluble (1% NaOH solution)	27.5
Alkali Soluble (18% solution)	41.3
Cation Exchange Capacity (mmol g^{-1})	2.39

Table 2. Results of extraction of 1 g coconut coir dust (of different particle sizes) in hot water.

Volume of Water (ml)	Particle Size (μm)	Mass of Residue (g)	Mass of Extract (g)	% Residue	% Extract
100	63	0.778±0.12	0.110±0.01	77.80	11.00
	75	0.752±0.04	0.08±0.04	75.20	8.00
	150	0.830±0.05	0.131±0.05	83.0	13.10
	212	0.799±0.02	0.165±0.02	79.90	16.50
	300	0.762±0.01	0.148±0.01	76.20	14.80
	425	0.901±0.01	0.043±0.01	90.10	4.30
	600	0.816±0.03	0.137±0.01	81.60	13.70
	850	0.981±0.02	0.107±0.02	98.10	10.70
200	63	0.750±0.01	0.180±0.01	75.0	18.0
	75	0.791±0.02	0.190±0.02	79.10	19.0
	150	0.765±0.05	0.196±0.05	76.50	19.60
	212	0.755±0.02	0.183±0.02	75.50	18.30
	300	0.746±0.01	0.185±0.01	74.60	18.50
	425	0.780±0.01	0.131±0.01	78.0	13.10
	600	0.831±0.03	0.151±0.01	83.10	15.10
	850	0.762±0.02	0.148±0.02	76.20	14.80
400	63	0.725±0.01	0.270±0.02	72.50	27.00
	75	0.770±0.02	0.211±0.02	77.0	22.10
	150	0.850±0.02	0.119±0.02	85.0	11.90
	212	0.740±0.02	0.211±0.016	74.0	21.10
	300	0.698±0.015	0.201±0.02	69.80	20.10
	425	0.805±0.01	0.178±0.01	80.05	17.80
	600	0.753±0.03	0.193±0.01	75.30	19.30
	850	0.807±0.02	0.121±0.02	80.70	12.10

was in the order of 400 ml > 200 ml > 100 ml. This may be attributable to the fact that coir dust is a biopolymer in which solubility of polymers involves two stages, swelling and disintegration (Billimeyer, 1980); and these two stages need large quantity of solvent. It may also be due to the nature of coir dust which is highly hydrophilic.

The variations of amount of extract and residues obtained from extraction of coir dust with its masses of three particle sizes (150, 300, and 850 μm) in 100 ml of water are shown in Figure1a and b. The amount of residue was greater than that for extracts for all the masses. The extracts were dependent on the masses of the coir dust; the smaller the mass, the higher the amount of extract. This is attributed to the fact that coir dust is highly hydrophilic with high water adsorptivity; absorbing about eight times its mass of water (Hume, 1949; Meerow; 1994). As the masses increased with no corresponding increase in quantity of solvent, there is less solubilization which results in low extracts.

Figures 2a and b show the result of extraction of different masses of coir dust of particle sizes ranging from 63–850 μm using acetone. As expected, smaller particle sizes yielded more extract than larger ones. Sze-Tao *et al.* (2001) reported an increase in quantity of extracts with decreased particle sizes for the extraction of tannins from walnut (*Juglans regia L.*). Similar results on the effect of particle sizes on the amount of extracts have been documented (Johnson *et al.*, 1978; Kim *et al.*, 2001).

The effect of different solvent systems, namely acetone/water (70/30), acetone/water (50/50), water and acetone, are shown in Table 3. Acetone/water (70/30) was found to be the best solvent system as it extracted the highest quantity (20%) from 1 g of coir dust while the least was acetone/water (50/50), extracting only 4.2%. Several workers have reported the dependence of efficiency of extraction on the type of solvent system used (Deshpande and Cheryan, 1985; Deshpande *et al.*, 1986; Pansera *et al.*, 2004). Giner-Chavez (1996) and Pansera *et al.* (2004) have reported acetone/water (70/30) as the best solvent system for extraction of tannins in tropical forages. The high efficiency of this solvent combination for the extraction of coir dust may be attributed to an optimum level of interplay of the properties of the two solvents – water and acetone at this combination. The properties of solvents that play significant role in dissolution of solutes include polarity, dielectric constant, polarizability, polarizing power, and hydrogen bonding (Pansara *et al.*, 2004).

Water is a polar aprotic solvent with high dielectric constant and boiling point, while acetone is non-polar protic

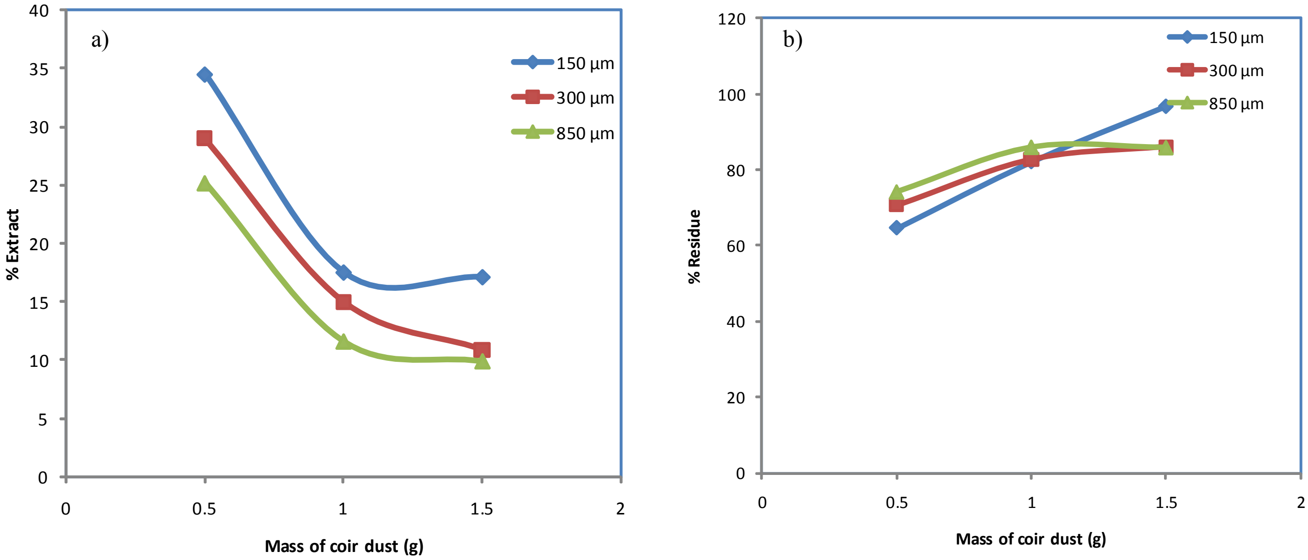

Figure 1. a) Variation of extracts in coir dust with mass at different particle sizes. b) Variation of residue in coir dust with mass at different particle sizes.

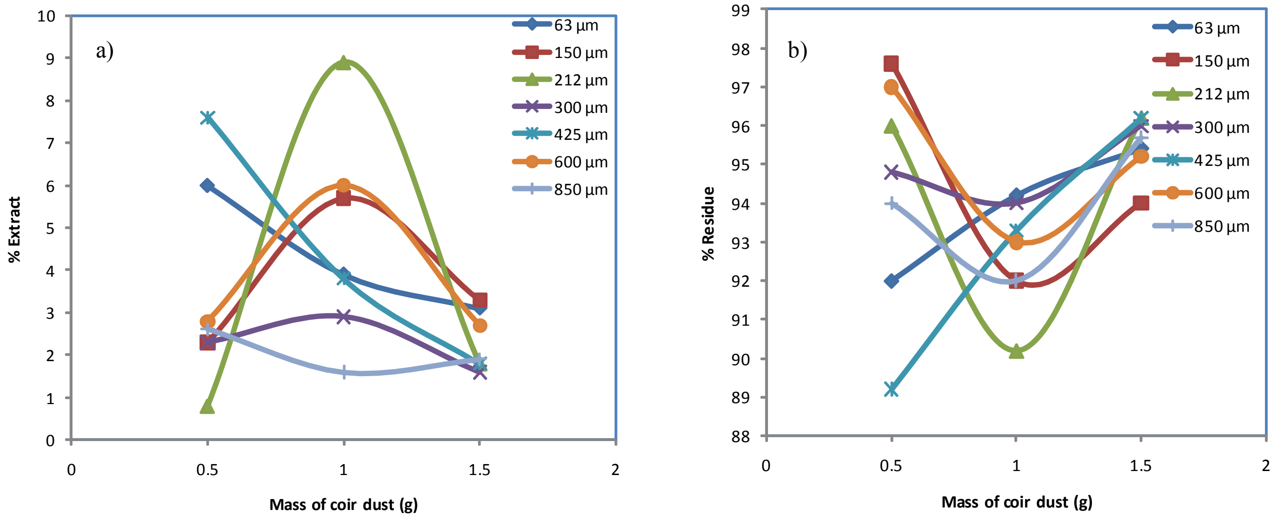

Figure 2. a) Variation of extracts in coir dust with mass at different particle sizes using acetone in soxhlet extractor. b) Variation of residue in coir dust with mass at different particle sizes using acetone in soxhlet extractor.

solvent with low dielectric constant having no hydroxyl groups. These properties affect the extent of mixing of solvent and solutes, which in turn determines the extent of solvation by the solvent. Water solvates negative species in the solutes through hydrogen bonding while acetone CH_3-C(=O)-CH_3 with its large dipole solvates only positively charged organic species. Water extracts through hydrosolubilization and hydrocracking of the sugar components in the coir dust, hence there were more extracts from water than that from acetone.

Poor tannin and lipid extractions with acetone from black acacia (*Acacia mearnsii*) and walnut (*Juqlans regia L.*) have been reported (Gonzalez-Vila *et al.*, 2000; Sze-Tao *et al.*, 2001; Pansera *et al.*, 2004). Acetone solubilized only the non-polar organic compound present in the coir dust while water solubilized both polar and non-polar components, hence there were less extracts in acetone. The least amount of extracts obtained from acetone-water (50/50) may be due to the low level of interaction of solute/solvent properties with the coir dust at this solvent combination. This is yet to be investigated.

Phytochemical screening of acetone extract of the coir dust has revealed the presence of active ingredients such as tannins, flavonoids, polyphenols, and phlobatannins, but

Table 3. Extract of 1 g of coconut coir dust (150 μm) using different solvent mixtures

Solvent (100 ml)	Mass of extract	% Extract
Water	0.131±0.05	13.1
Acetone	0.066±0.001	6.60
Acetone/Water (70/30)	0.20±0.001	20.0
Acetone/Water (50/50)	0.042±0.001	4.20

absence of anthraquinones and saponnins (Table 4). The presence of tannins, which are polyphenols, have been reported in coir dust (Tejano *et al.*, 1985; Joseph and Sarma, 1997). Tannins are major polyhydroxy compounds extractable from plant materials, barks, fruits, leaves, seeds, roots using polar, non-polar or mixed solvent systems. They are not pure chemical compounds with known structures but they contain numerous components with phenolic moieties, such as catechol and pyrogallol condensed into a complicated polymeric structure (Kantz and Singleton, 1991).

4. Conclusions

The characterization and extraction of coconut (*Cocos nucifera L.*) has shown that it has both solid and extractable contents. The solid contents, mostly cellulose and lignins, constitute above 90% of its composition while the extractives mainly polyhydroxy compounds (tannins, hemicelluloses, pectins) combined together are below 10% of its total content. Coir dust can therefore be a potential adsorbent/ion exchanger as a result of its constituents (lignin, cellulose, and extractives) bearing polyhydroxy groups which can sorb or exchange ions from surrounding solution.

The best solvent system for extraction of coir dust was found to be acetone/water (70/30). Thus coconut coir dust, a once discarded and underutilized agricultural waste, can be of great value in industrial and other scientific applications.

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Table 4. Phytochemical screening of acetone extract of coconut (*Cocos nucifera L.)* coir dust.

Test	Observation	Inference
Tannins		
10ml of acetone extract filtrate + few drops of $FeCl_3$ reagent.	Blue-black precipitate	Tannins present
5 drops of filtrate from acetone extract + 10ml of deionized water + 3 drops of bromine water	Brownish colour of filtrate was decolorized	Tannins confirmed
Flavanoids		
Few pieces of magnesium ribbon + 5ml of filtrate + 3 drops of conc. HCl	Red coloured solution	Flavanoids present
Polyphenols		
1 ml of filtrate from acetone extract + 2 drops of mixture of 1 ml 1% $FeCl_3$ solution with 1ml of 1% Potassium Ferricyanide	Green-blue coloration	Polyphenols present
Phlobatannins		
2 ml of filtrate from acetone extract + 1 ml dil. HCl and boiled for 2 minutes in a test tube.	Pale yellow colour	Phlobatannins present
Anthraquinone		
2 ml of filtrate from acetone extract +2 drops of 3% NH_4OH	No red coloration	Anthraquinone absent.
Saponnins		
Frothing test: 1 ml of filtrate from acetone extract shakened vigorously	No formation of lather	Saponnins absent.
Emulsion test: Two drops of olive oil was added to the mixture in frothing test above	No emulsion formed.	Saponnins absent.

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