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Effect of repeated extraction of white ash with water on physicochemical properties of *Palash Kshar*

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Kshar Kalpana is the method of extraction of white alkaline substances present in the white ash of the plant. Ancient scholars used single- time extraction by variation in the quantum (4, 6 and 8 times) of solvent (water). Modern pharmaceutics recommend the repetition of extraction till getting maximum extract. So it is necessary to compare the *Palash Kshar* prepared by using single and repeated extraction by using water. *Palash Kshar* was prepared by using single and repeated (5 times) extraction of white ash of *Palash* (*Butea monosperma* Lam.) along with water. *Palash Kshar* was characterized by organoleptic characters, physico-chemical limits and various modern analytical techniques, viz., X-Ray Diffraction (XRD), Energy Dispersive X-Ray Spectroscopy (EDS) and Flame Photometry. The percentage yield of *Palash Kshar* decreased nearly half from 1st to 5th extraction. Maximum Sodium (Na) and potassium (K) extraction happens after the 1st extraction. The K percentage of *Palash Kshar* obtained after 1st, 2nd, 3rd and 4th extractions is 5.380, 6.177, 5.959, and 3.990. Na percentage of *Palash Kshar* obtained after 1st, 2nd, 3rd and 4th extractions is 0.179, 0.207, 0.329 and 0.321. All samples of *Palash Kshar* contain K, Na, C, O, Cl, while Mg and Al were present in *Kshar* obtained from repeated extraction. A method prescribed by ancient scholars i.e., the use of a single extraction of ash with water, is the most appropriate method in terms of yield, cost, and extraction of basic substances like Na, K.

Keywords: *Palash Kshar*, Potassium, Sodium

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Bhaishjya Kalpana deals with the conversion of raw herbs into different formulations. Conversion of raw drugs into formulation is done by using drugs as a total [*Kalka* (paste), *Churna* (powder), *Mashi* (black ash) and *Vati* (tablet)], or extracting its useful part and by a combination of other substances like *Ghruta* (clarified butter), sugar with whole or extracted raw drug. *Swarasa* (juice), *Kwatha* (decoction), *Ksheerapaka* (decoction with milk), *Hima* (cold infusion), *Phanta* (hot infusion), *Arka* (volatile extract), *Satva* (starchy extract), *Ghana* (solid water extract) and *Kshar* are the formulation where the useful part of the plant is extracted. Ancient scholars used different methods of extraction by varying type of solvent (cold, hot water), types of solvent (*Ksheerpaka*, *Snehapaka*, *Sandhana Kalpana*) and different applications like heat.

Kshar is an imperative treatment tool as it has dominance over all surgical and para surgical measures. As per *Acharya Vagbhat*, diseases which are difficult to cure can be treated by *Kshara* therapy. *Kshar Kalpana* (white colored residue obtained after evaporation of filtrate of the plant ash dissolved in water) deals with extraction of water-soluble material from white ash of the plant. *Kshar Kalpana* s mainly prepared through five unit operations: 1. Open pan burning, 2. Maceration of ash with specific quantum of water, 3. Sedimentation, 4. Filtration, and 5. Evaporation of filtrate[1]. Quality of finished product mainly depends upon of quality of raw material and different process used in the method of preparation. Variation in any of the above five steps of *Kshar* preparation may alter the quality of *Kshar* obtained.

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Abbreviations:

PK 1: *Palash Kshar* obtained after 1st extraction of white ash of *Palash* with water.

PK 2: *Palash Kshar* obtained after 2nd extraction of white ash of *Palash* with water.

PK 3: *Palash Kshar* obtained after 3rd extraction of white ash of *Palash* with water.

PK 4: *Palash Kshar* obtained after 4th extraction of white ash of *Palash* with water.

PK 5: *Palash Kshar* obtained after 5th extraction of white ash of *Palash* with water.

Maceration of ash with a specific quantum of water (Step 2), Sedimentation (Step 3) and Filtration (Step 4) is done to extract water-soluble part from the white ash of the plant. Ancient scholars used single-time use of water for the extraction of water-soluble parts from the white ash of plants. But the extraction process can be repeated till the completion of extraction to increase the yield.

An ancient scholar used *Palash Kshar* in the *Gulma, Plihavruddhi* (splenomegaly), *Yakrut vruddhi* (hepatomegaly), *Ashmari* (renal calculi), *Shukra granthi* (more viscous seminal fluid)[2]. *Palash Kshar* is used as antihelmintic, antiimplantation, antiovulatory, abortificient, antilaprotic, antigout, antiestrigenic, spasmogenic, antifungal, antispasmodic, hypotensive, astringent, alterative, aphrodisiac, antiasthmatic, and bactericidal[3]. Few research works are conducted on validation and physico-chemical variation in *Kshar* by varying concentrations of solvent (water) and plant variation[4-6]. Also few research works are conducted on physico-chemical properties of *Palash Kshar*[7,8].

However, no single work is conducted to study pharmaceutical and physicochemical variation on single and repeated extraction of alkaline substances from the white ash of a *Palash* (*Butea monosperma Lam.*) plant using water. So, the present study is planned to extract *Palash Kshar* by using a repeated extraction method till no or less extract is obtained. The *Kshar* obtained by using single and multiple extractions is compared with different physicochemical parameters, cost and yield.

The aims and objectives of the present investigation are as follows: 1. to prepare *Palash Kshar* by using single and multiple extractions with water from white ash of *Palash* plant, and 2. to compare *Palash Kshar* by using single and multiple extraction in terms of physicochemical parameters, cost and yield.

Material and Methods

Preparation of the ash of *Palash* plant

About 11.890 kg of white ash was obtained after burning of 66.550 kg of the whole *Palash* plant in an open field.

Preparation of *Kshar jala*

Kshar jala was prepared by using 4 times of water for dissolution of alkaline material from ash into the water[1].White ash remains after first extraction was once again extracted with 4 times of water. The above procedure was repeated for another three times. Three batches of each method were prepared. The white ash was rubbed with water till the pH of water remains constant and allowed to settle for 12 h. The *Kshar jala* was obtained after filtration by using three folded cotton cloth. RO water was used to avoid more alkaline material from hard water. 60 liter capacity aluminum (Al) vessel with height 310 mm and 505 mm diameter was used for mixing of ash and water. 11 liter capacity Al vessel with height 260 mm and 280 mm diameter was used for evaporation of *Kshar jala*. A rubber tube of 1 inch diameter and 266 cm length was used for removing supernatant *Kshar jala*. White ash is macerated with water till the pH of water remains constant.

Preparation of *Palash Kshar*

Palash Kshar was obtained by evaporating the water content from the filtrate (*Kshar jala*) in an open pan vessel.

Physicochemical analysis

Loss on drying[9], water soluble extractive value[10], total ash value[11], density[12], determination of pH using pH-meter[13], determination of percentage of sodium (Na) and potassium (K) by flame photometer[14] was calculated using methods mentioned in Ayurvedic pharmacopeia. X-ray diffraction **(**XRD) and Energy dispersive x-ray spectroscopy (EDS) of all samples of *Palash Kshar* was also done.

Results

Preparation of *Palash Kshar*

The details of *Palash Kshar* preparation are given in Table 1 and 2.

The average increase in pH after 1st, 2nd, 3rd, 4th and 5th maceration of ash with water is 5.5, 3.36, 3.03, 2.66 and 2.33 respectively. The time required for maceration of ash with water is in the range of 45 to 53 min. Three to four times filtration is required after average 16 hrs. of sedimentation to get clear fluid. There is an increase in yield of *Kshar jala* after 2nd, 3rd, 4th and 5th extraction than 1st extraction.

Yield of *Palash Kshar*

The details of the yield of *Kshar* after repeated extraction with water are given in Graph 1.

There is a decrease in the percentage of the yield of *Kshar* to near about half after each repeated extraction. But there is double in percentage of *Kshar* after all five times extraction with water than the single time extraction with water.

Table 1 — Details of *Palash Kshar* preparation after 1st, 2nd, 3rd, 4th and 5th extraction with water

Sr. No.	Parameters	PK1	PK 2	PK 3	PK 4	PK 5
1	White ash quantity (kg)	3.4	3.4	3.4	3.4	3.4
2	Avg. pH of water	6.06	6.66	6.53	6.43	6.76
3	Avg. pH of *Kshar jala* after maceration	11.56	10.03	9.56	9.03	9.1
4	Time required for maceration till pH remain constant (min)	50	53.33	51.66	45	48.33
5	Time given for sedimentation (h)	15.43	17.26	16.40	16.15	16
6	Details of filtration	4 folded cloth	4 folded cloth	4 folded cloth	4 folded Cloth	4 folded cloth
7	Frequency of filtration	3 time	3 time	3 and 4 time	3and 4 times	3 and 4 time
8	Avg. pH of *Kshar jala* after filtration	11.33	10.2	9.7	9.26	9.2
9	Filtration time (hr)	1.10	1. 40	1.25	1.48	1.40
10	Avg. quantity of *Kshar jala* obtained (l)	9.4	10.21	11.4	10.03	10.16
11	Avg. Yield of *Kshar jala* in %	69.11	75.11	83.81	73.75	74.75
12	Color of *Kshar jala*	Yellowish Green	Yellowish Green	Greenish white	White	Greyish white
13	Weight of remaining wet ash (g)	6698	5703.66	5627.66	5429	5246
14	Weight of *Kshar* obtained (g)	269.66	111	44.5	16.66	7
15	Yield in percentage	7.92	3.26	1.3	0.48	0.20
16	Total Potassium extraction (g)	14.4184	6.300	2.8305	0.6647	---
17	Total Sodium extraction (g)	0.4797	0.2111	0.156	0.0534	---

Table 2 — Observation made during preparation of all samples of *Palash Kshar*

Sr. No.	Time	Observations				
		PK 1	PK 2	PK3	PK4	PK5
1	Initial (room temp.)	24- 30 ^{0}C	28-30^0 C	24-28^0 C	26-30^{0}c	30 ^{0}c
2	0-30 min.	94- 96 ^{0}C Boiling started	90-92^{0}C Boiling started	94-95^{0}C Boiling started	88- 94^{0}C Boling started	98^{0}C Boling started
3	30-60 min.	100^{0}C Vigorous boiling	100^{0}C Vigorous boiling	100^{0}C Vigorous boiling	100^0 C Vigorous boiling	100 ^{O}C Vigorous boiling
4	60- 90 min.	100^{0}C	100^{0}C	100^{0}C	100^0 C	100^0 C
5	90-120 min.	100^{0}C	100^{0}C	100^{0}C	100^0 C	100^{0}C
6	120-150 min.	100^{0}C	100^{0}C	100^{0}C	100^0 C	100^{0}C
7	150-180 min.	100^{0}C Whitish substance start to adhere	100^{0}C Whitish substance start to adhere	100^{0}C Whitish substance start to adhere	100^0 C Whitish substance start to adhere	100 ^{0}C Whitish substance start to adhere

Cost of *Palash Kshar*

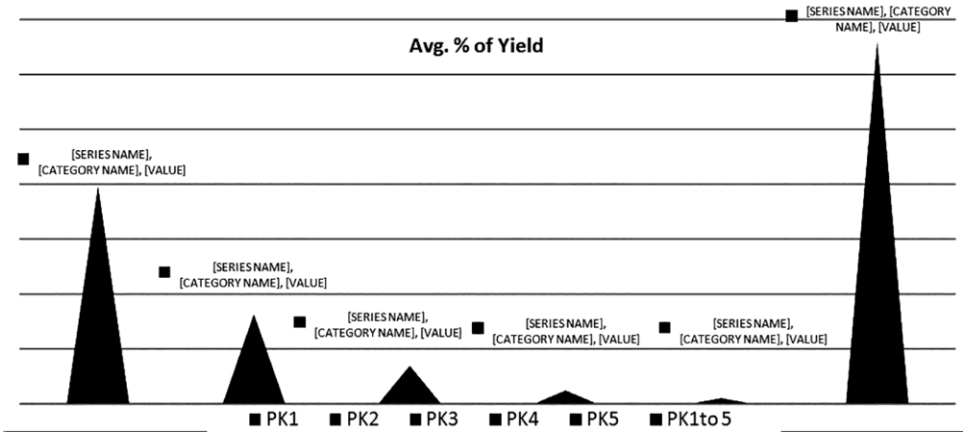
The details of the production cost of *kshar* after repeated extraction with water is given in Graph 2. PK 3, PK 4 and PK 5 were costlier than PK 1and PK 2.

Results of physico-chemical parameters, Flame Photometry, are presented in Table 3. Results of EDS are presented in Table 4.

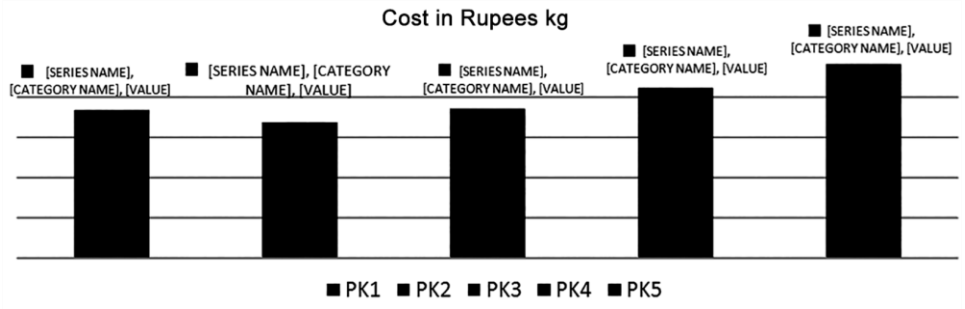
Physicochemical parameters of *Palash Kshar* obtained after repeated extraction are within the limits mentioned in the API except for the water-soluble extractive of *Kshar* obtained after 1st extraction. Water-soluble extractive is slightly less than API values. XRD patterns of different samples of *Palash Kshar* are given in Figures 1 to 4. The XRD pattern of all samples showed all major peaks and minor peaks at the same 2θ values. This shows that crystals present after all extractions are near about the same.

Discussion

Kshar Kalpana is mainly derived after evaporation of water soluble contents from white ash. Solubility of ash is a function of the amount of alkali metal components (hydroxides or carbonates of Na and K) and other soluble salts (like chlorides and sulfates of K and Na) present in the ash. *Mrudu* (Mild) and *Madhyam* (moderate) *Kshar* are recommended internally, while *Tikshna* (strong) *Kshar* are used externally. Types of *Kshar* depend upon pH and concentration of alkalis. More concentration of potassium and hydroxide/ chloride form makes *Kshar* stronger.



Graph 1 — Yield of *Palash Kshar* after repeated extraction



Graph 2 — Production cost of *Palash Kshar* after repeated extraction

Table 3 — Physicochemical parameters and sodium and potassium percentage of *Palash* ash and different samples of *Palash Kshar*

Test	Standards[15]	*Palash* Ash	PK 1	PK 2	PK 3	PK 4
Appearance	Fine Powder	Fine powder	Fine Powder	Fine powder	Fine powder	Fine powder
Color	Greyish White	Dark grey	Greyish White	Grey	Greyish White	Greyish White
Odor	Burnt smell	Burnt smell	Slightly Burnt smell	Slightly Burnt smell	Slightly Burnt smell	Slightly Burnt smell
Taste	Salty	Salty and Unpleasant	Salty	Salty and Unpleasant	Salty and Unpleasant	Salty and Unpleasant
L.O.D. (%)	Below 5	3.78	2.01	2.01	2.64	1.58
Water Soluble Extract (%)	NLT 98	76.23	96.25	98.25	99.86	99.59
Ash Value (%)	NLT 65	67.86	91.25	91.25	89.67	90.25
pH	10-11	10.1	10.2	10.2	10.9	10.7
Density (g/mL)	0.70-0.9	0.89	0.84	0.84	0.86	0.88
Potassium (K) mg/100 g	----	5490	5380	6177	5959	3990
Sodium (Na) mg/100 g	----	180	179	207	329	321

Physicochemical parameters

The increases in pH of RO water after each extraction indicate the solubility of water soluble alkali from the ash. Decrease in pH after repetition of extraction is due to decrease in solubility of alkaline material from ash after repetition of extraction.

The decrease in *Kshar* yield may be due to a decrease in the potash solubility from ash into water. There is no need to do an extraction of *Kshar* after the 3rd extraction as it becomes costlier.

Flame photometry and EDS

Out of 186.66 g of K present in 3.4 kg of ash, only 24.50 g is extracted after a total 5 extractions, which is just 13.12%. Out of 6.22 g of Na present in 3.4 kg of ash, only 0.18 g is extracted after a total five

extractions which is just 2.89%. Maximum sodium (7.83%) and potassium (7.72%) extraction happens after 1st extraction and then there is a decrease in the extraction of Sodium (6.87%) and Potassium (5.24%) from 2nd to 4th extraction. An increase in potassium percentage is observed in PK 2 and PK 3 than in PK 1, while PK 4 has less potassium percentage than PK 1. An increase in sodium percentage is seen in PK 2, PK 3 and PK 4 rather than PK 1. This may lead to higher pH of PK 3 (10.7) and PK 4 (10.9) than PK 1(10.2). So PK 1 is mild alkaline in nature, which was recommended by ancient scholars for internal use to prevent damage or harm to mucous membranes.

Table 4 — Quantification of different elements using energy dispersive X-Ray spectroscopy (EDS)

Element	PK 1	PK 2	PK 3	PK 4
C	11.16	18.20	18.65	19.17
O	36.86	50.11	49.44	43.34
Na	1.00	0.86	1.44	1.10
Mg	ND	0.14	0.21	6.00
AL	ND	1.51	0.22	ND
CL	0.29	0.42	0.49	1.17
K	50.18	28.77	29.04	27.47
S	ND	ND	0.52	00.00
Mo	ND	ND	ND	1.76
	100.00	100.00	100.00	100.00

ND: Not Detected.

The carbon and oxygen element percentage is more in PK 2, PK 3 and PK 4 than PK 1, which may be due to increase in extraction of carbonate form of alkali metal after repeated extraction with water. Mg percentage is increased from PK 2 to PK 4, while Mg is not detected in PK 1. The presence of aluminum in PK 2 and PK 3 may be due to the use of an aluminum vessel for sedimentation. There is a need to evaluate the pharmacological action of *Kshar* to understand the role of these different elements.

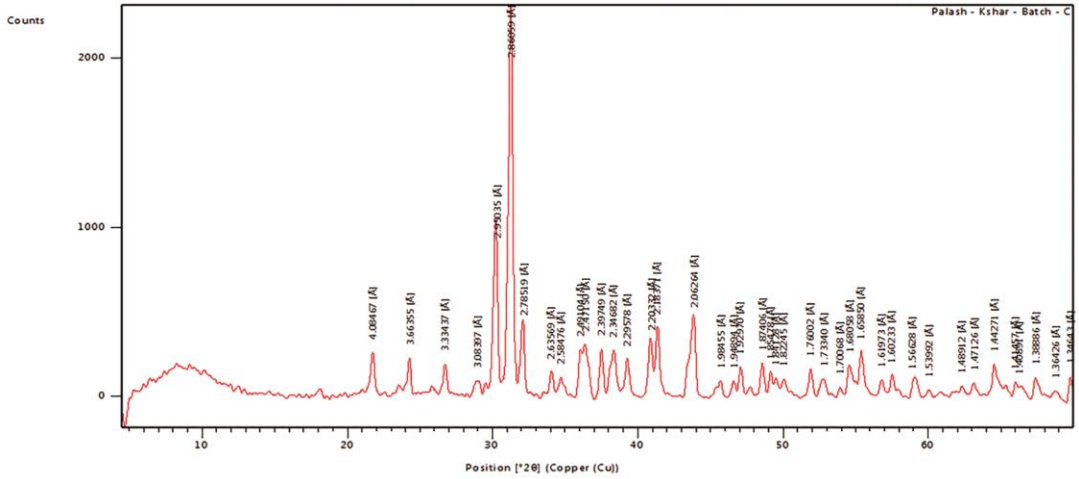


Fig. 1 — X ray diffraction pattern of *Palash Kshar* 1

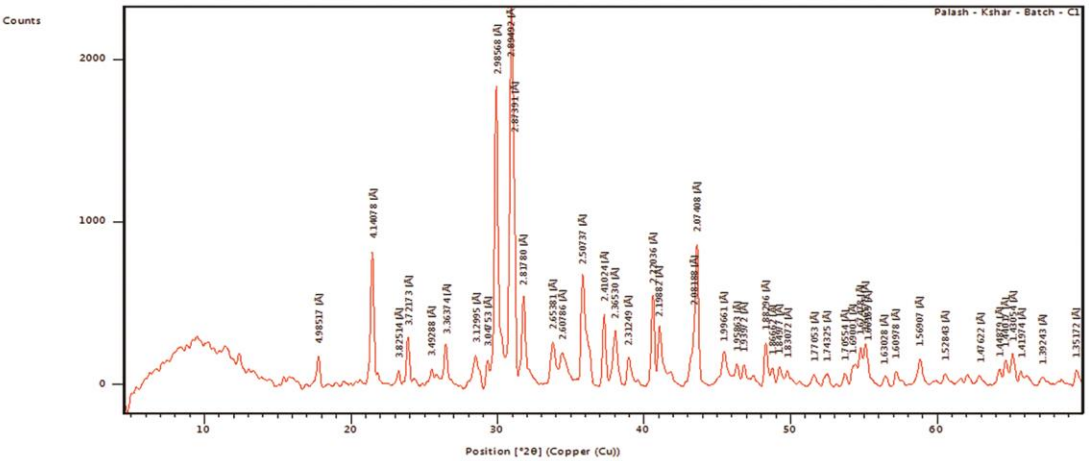


Fig. 2 — X ray diffraction pattern of *Palash Kshar* 2

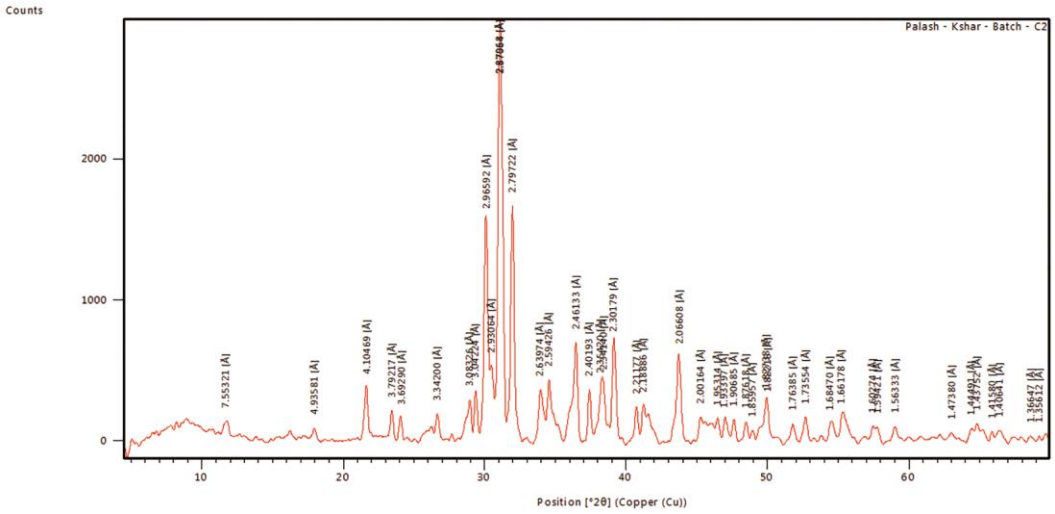


Fig. 3 — X ray diffraction pattern of *Palash Kshar* 3

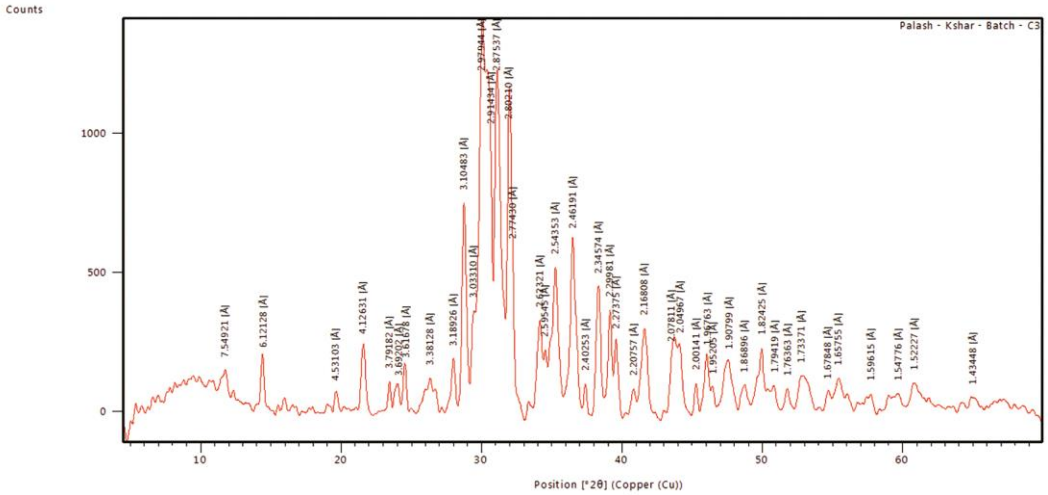


Fig. 4 — X ray diffraction pattern of *Palash Kshar* 4

XRD

XRD pattern of all samples showed all major peaks and minor peaks at the same 2θ values, which shows that the crystals present after all extractions are near about the same. No peak is comparable with the peaks of halite and sylvine crystals of NaCl and KCl. EDS reports show the presence of Na, K, Cl, C and O molecules in the *Palash Kshar*. This may indicate that Na and K may not present in chloride form, but may present in carbonate form. This can be supported by the presence of a greater percentage of the carbonate and sulfate form than the chloride form mentioned in other research articles[7,8].

Conclusions

Palash Kshar obtained after 5 repeated extractions increase the yield to double than single extraction, but it's costlier. Na and K are mainly present in carbonate form rather than chloride in *Palash Kshar*. Less pH, less N and K percentage, more yield and less cost justify the use of a single extraction method by ancient scholars than repeated extraction.

Conflict of Interest

Authors declare that there is no conflict of interest.

Authors' Contributions

PKG conceived and designed the study plan. VRN completed the literature search and acquired the data. VRN and PKG drafted the initial report, analysed and interpreted the data. PKG, CEL and RKI critically reviewed and finalized the manuscript. All authors have approved the final version and are in agreement to submit the manuscript.

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