



Physico-chemical Characterization of Siddha Herbomineral Formulation *Navachaara chunnam*

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ABSTRACT

Introduction: The Holistic Siddha system of medicine aims to reestablish the wellbeing of humans by understanding the fundamental causes of the illnesses. It endeavours to assault the root causes by detoxifying, cleansing, reinforcing body tissues (dhatus) and adjusting substantial doshas, guaranteeing total remedy. The Siddhas composed their information in palm leaf manuscripts, parts of which were found in distinctive parts of South India. They recorded various important solutions for each illness. Among that *Navachaara Chunnam* is one of the effective medicines for Liver related ailments. The main ingredients of this preparation include *Navachaaram* (ammonium chloride), lemon juice and *Uvar Mann* (*pooneeru mann*). All the materials were purified and the medicine was prepared according to the classical siddha text. This study aims to scientifically validate the Siddha preparation "*Navachaara Chunnam*" using Siddha and modern standardization procedures in order to improve Siddha medicine research. As a result, this paper provides evidence to support the efficacy of this Siddha medication.

Aim & Objective: To estimate the physico-chemical and instrumental analysis XRD and PSA for navachaara chunnam.

Materials and Methods: The formulation was prepared as per the exemplary siddha literature. The physico chemical analysis such as Loss on Drying, Total Ash, Water Soluble Ash, Acid Insoluble Ash, Water Soluble Extractive, Alcohol Soluble Extractive and pH were carried out. The instrumental analysis such as XRD and PSA were also carried out.

Results: The physico chemical analysis revealed that the Loss on Drying was 0.64%, Total Ash was 97.90%, Water Soluble Ash was 23.12%, Acid Insoluble Ash was 33.69%, Water Soluble Extractive was 32.64%, Alcohol Soluble Extractive was 16.64% and pH was 6. The chemical characterization of *Navachaara Chunnam* was also evaluated by XRD and PSA methods.

Keywords: Physico-chemical, Instrumental Analysis, Xray Diffraction, Particle Size Analysis, *Navachaara chunnam*.

INTRODUCTION

The Siddha system of medicine has been used to treat a variety of illnesses holistically since ancient times¹. Siddha formulations are made from the crude drugs that are available as natural herbs and also as Mineral and in Metal form¹. The primary motto of Siddha is "Food should be taken as medication or probably medication should be taken as food. These sorts of medications are effectively and promptly available. The normalization of a medication is extremely fundamental and compulsory requirement, with the goal that the nature of medicine can be accomplished according to the norm of present-day scientific culture. Despite the fact that numerous Siddha formulations have been used to treat a variety of diseases, there is a push for global acceptance due to a lack of scientific validation and documentation. Modern methods can be used to overcome the limitations and ensure the quality, safety, and therapeutic efficacy of Siddha formulations.

Standardization is a vast area that can be covered by evaluating the medication in accordance with the necessary modern standardization guidelines. It is a part of the process of developing drugs¹. Before a Siddha formulation is used in *in vivo*, *in vitro* or clinical trials, it is essential to determine its quality¹. Generally, crude drugs

are submitted to series of processes like purification, trituration, incineration and calcination to get the end product². The drugs' quality control will be evaluated by standardizing them. Standardization of drug is crucial for exhibiting its identity purity and quality assurance. Therapeutic plants and minerals are helpful in treating various liver related ailments yet there is a pre-requisite of standardization to figure out its viability. *Navachaara chunnam* is one of the herbo - mineral formulation referenced in Siddha literature for the treatment of hepato-splenomegaly. Although such countless conventional drugs are accessible for diseases like in modern system of medicine, in general people are looking for natural drugs because of their safety and viability.

The various constituents present in Herbo-mineral drugs play a multitarget and synergistic job in the treatment of Anaemia, Hepatosplenomegaly. Calcinated nanoparticle powder or chunnam is one of the 32 varieties of internal medicine found in Siddha³. Metals, minerals, and marine products that constitute a Chunnam preparation. Being a higher-order drug, Chunnam has more therapeutic benefit even at smaller dosages⁴. Thus, this study is going to analyse the drug *Navachaara chunnam* in siddha and modern standardization techniques.



OBJECTIVES

The aim of the study is to do Physico-chemical and Preliminary instrumental analysis for the drug *Navachaara chunnam*.

MATERIALS AND METHODS

Collection and Identification of Drugs:

The herbs and minerals used for the formulation were identified and authenticated by faculty of Department of Gunapadam and Botanist of National Institute of Siddha, Chennai – 47.

Ingredients of *Navachaara Chunnam*:

Purified *Navachaaram* (Ammonium Chloride)

Lemon juice

Uvar mann (Pooneeru)

Preparation of Drug:

The purified *Navachaaram* was ground with lemon juice for 12 hrs to make villai. The villai was made to dry. A pit of breadth and height 2.5cm*2.5 cm was dug in which the half of the pit was filled with pooneeru and the villais were placed over it and it was again covered with pooneeru up to the edge of the pit. The dried cow dung of 10500 gms weight were placed over the pit for incineration process and allowed to cool. The prepared drug was stored in an airtight container.

Administration:

- **Dosage:** 2 - 7 kundrimani edai (260-910 mg /kg)
- **Adjuvant:** Sombu Theneer, Neermulli Samoola Kudineer
- **Indication:** Vallaikatti (Hepatomegaly), Kavusaikatti, KendaiKatti, Magodharam (Ascites), Neerambal, Paandu (Anaemia), Soothaga katti (Uterine disorders)
- **Route Of Administration:** Oral.

Methodology:

All the physicochemical parameters were carried out as per the standard test procedures (Protocol for testing of Ayurvedic, Siddha and Unani medicines. Ghaziabad: Government of India, Department of AYUSH, Ministry of Health & Family Welfare, Pharmacopeial Laboratory for Indian Medicines.

Standardization as per Siddha Classical Literature

Standardization of a drug means confirming its purity, quality and identifying any adulterants.

The test drug's organoleptic characters were evaluating the Colour, Taste, Odour, Flow Property, Lustre, Fingerprint Test, Texture⁴. Particle Size of 1gram of the test medication were evaluated in daylight with naked eye⁴. The results were noted.

Standardization as per Modern Aspect

Physico-Chemical Analysis

Physico-chemical analysis relies on a wide variety of analysis techniques to know the intrinsic properties of molecules or atoms. The following Physicochemical studies of the trial drug have been done according to the PLIM guidelines.

1. Loss of Drying

5g of the test drug NC was accurately measured and taken in a tared evaporating dish. The test drug was placed in an oven and heated at 105°C for five hours. The procedure is continued until the difference between two consecutive procedure is not more than 0.25 percent. Then the percentage of moisture content in the sample was calculated in comparison with shade dried drug.

2. Determination of Total Ash

2g of the test drug was accurately measured and taken on tared silica dish. The test drug was placed on the furnace and incinerated at 450°C until it is carbon free. The sample is left to cool and weighed. The percentage of total ash is calculated in comparison with the air-dried drug.

3. Determination of Acid Insoluble Ash

25ml of hydrochloric acid was added to the ash and the insoluble matter was collected. It was washed with hot water. Then the sample was dried on hot plate and ignited until it reaches a constant weight. It is then left too cool in a desiccator for 30 minutes and weighed. Then the acid insoluble ash was calculated with reference to air dried drug.

4. Determination of water-soluble ash

2g of the ash was boiled in 25 ml of water for 25 minutes, the insoluble material was collected in crucible. The collected material is washed with hot water and boiled ignited for five minutes at 450 °c. the weight of the insoluble matter was subtracted from the weight of ash. This represented the water-soluble ash and the percentage of water-soluble ash was calculated with reference to that of the air-dried drug.

5. Determination of Water-Soluble Extractive

5gms of NC was macerated with 100ml of chloroform water in a closed flask for 24 hours, shaken frequently for 6 hours and it was allowed to stand for eighteen hours. The solution was filtered rapidly with taking precautions to prevent loss of solvent. 25 ml of the filtrate was evaporated to dryness in a tarred flat bottom shallow dish, further dried at 105°C to constant weight and weighed. The percentage of water-soluble extractive was calculated with reference to the air-dried drugs.

6. Determination of Alcohol Soluble Extractive

The test drug NC was macerated with 100ml of alcohol in a closed flask for twenty-four hours, shaken frequently for six hours and it was allowed to stand for eighteen hours.



The solution was filtered rapidly with taking precautions to prevent loss of solvent. 25 ml of the filtrate was evaporated to dryness in a tarred flat bottom shallow dish, further dried at 105°C to constant weight and weighed. The percentage of alcohol soluble extractive was calculated with reference to the air dried drug.

XRD Analysis:

Powder diffraction data were collected by Aeris PANalytical diffractometer (Netherlands) with Ni-filtered copper radiation in Bragg-Brentano geometry. Fine powder of the sample taken in a thin layer on a silicon zero background holder. The sample was recorded for the angle 2θ in the range of 10-90 degrees at a scanning rate of 4 degrees/sec with $\text{CuK}\alpha$ (λ 1.5418 Å). The XRD pattern of Navachaara Chunnam reveals the presence of several crystalline phases. The main peaks in the XRD pattern correspond to specific d-spacings, which are indicative of different mineral components.

Particle Size Analysis:

Particle size of two different formulations were determined by using JINAN WINNER 2000E particle size analyser after suspending a small amount of formulation in aqueous dispersing phase and stirring it for 5 min on a magnetic stirrer at room temperature. Microscopic obedience of the particle dimensions examination indicates that the mean intermediate particle dimension of the sample was sized up to be 41.707 μm from the raw drug particle dimension which was about 66.532 μm and so ensures the solubility, processing belongings, bioavailability, product uniformity, strength and the medicinal result of the trial drug NC.

RESULTS

XRD Pattern: Counts

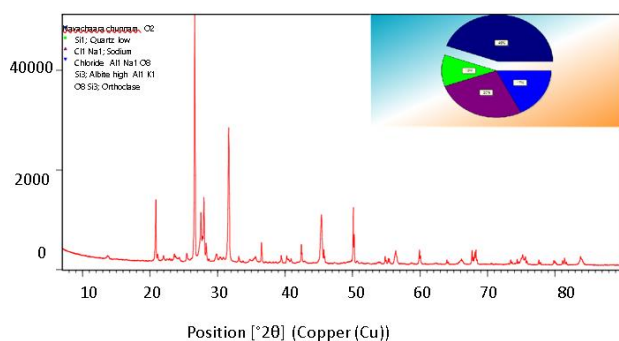


Figure 1: XRD Main Graphics and Analysis View

Peak List of XRD:

Table 1: Peak list of XRD

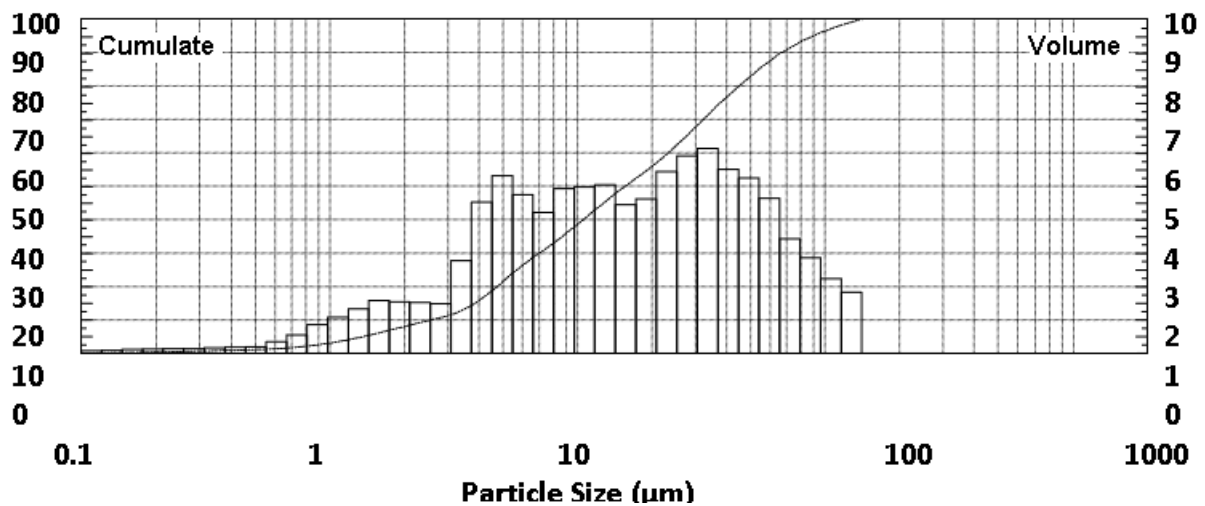
Pos. [2θ]	Height [cts]	FWHM Left [2θ]	d-spacing [Å]	Rel. Int. [%]
13.7192	294.82	0.3078	6.44945	0.78
20.8174	9156.54	0.1054	4.26361	24.17
21.1060	829.45	0.1076	4.20596	2.19
21.9660	820.41	0.1220	4.04319	2.17
22.6628	231.30	0.7101	3.92042	0.61
23.5750	853.04	0.1288	3.77076	2.25

Pattern List:

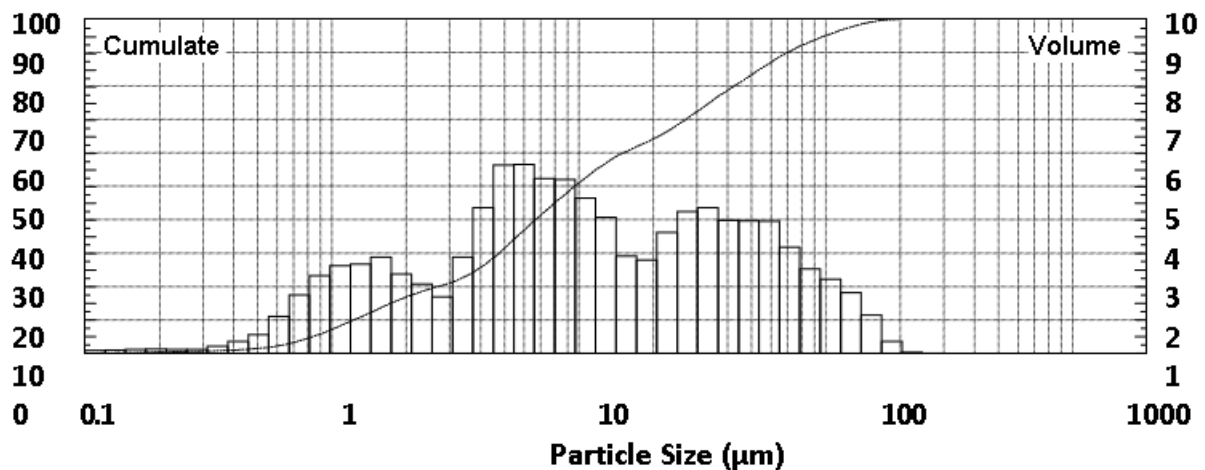
23.9836	455.44	0.8071	3.70743	1.20
25.4255	1232.70	0.1451	3.50036	3.25
26.5877	37882.26	0.1059	3.34992	100.00
27.5122	6647.37	0.2073	3.23942	17.55
27.9582	9126.27	0.1152	3.18875	24.09
28.3016	2660.79	0.0950	3.15083	7.02
28.7094	478.21	0.0865	3.10700	1.26
29.7925	1097.42	0.2933	2.99646	2.90
30.3577	542.51	0.2626	2.94195	1.43
30.8340	505.26	0.3200	2.89758	1.33
31.6142	19606.39	0.1789	2.82783	51.76
33.1267	923.14	0.1167	2.70209	2.44
34.7774	396.20	0.2220	2.57752	1.05
35.5096	704.25	0.5347	2.52604	1.86
36.5041	3620.56	0.0852	2.45947	9.56
39.4174	1311.11	0.1078	2.28414	3.46
40.2588	1173.96	0.1106	2.23832	3.10
40.7844	440.15	0.2547	2.21068	1.16
42.4148	3279.84	0.0958	2.12940	8.66
45.2652	4300.95	0.1742	2.00171	11.35
45.3882	5611.30	0.1486	1.99657	14.81
45.7602	2249.74	0.0803	1.98120	5.94
50.1050	10902.48	0.0807	1.81911	28.78
50.6569	428.78	0.1549	1.80058	1.13
53.8452	297.57	0.1770	1.70124	0.79
54.8249	1380.25	0.1029	1.67314	3.64
55.2879	1217.77	0.0786	1.66021	3.21
56.3475	1797.26	0.3068	1.63148	4.74
58.7342	169.20	0.0917	1.57075	0.45
59.9123	2778.35	0.1148	1.54264	7.33
62.2598	160.05	0.1175	1.49000	0.42
63.9833	815.81	0.1165	1.45395	2.15
66.0495	591.85	0.5297	1.41339	1.56
67.6989	2617.17	0.1107	1.38291	6.91
68.1018	2264.80	0.1234	1.37571	5.98
68.2719	1712.58	0.1084	1.37270	4.52
73.4300	910.88	0.1030	1.28848	2.40
74.3857	863.69	0.0743	1.27428	2.28
75.1923	1450.35	0.3339	1.26260	3.83
77.6161	903.22	0.1128	1.22911	2.38
79.8354	812.93	0.1493	1.20043	2.15
81.1344	828.57	0.1170	1.18446	2.19
81.4333	1114.97	0.1256	1.18086	2.94
83.7896	1394.81	0.2781	1.15355	3.68

Table 2: Pattern list of XRD:

Visible	Ref.Code	Score	Compound Name	Displ.[°2θ]	Scale Fac.	Chem. Formula
*	98-006-2404	60	Quartz low	0.000	1.010	Si ₁ O ₂
*	98-016-5592	45	Sodium Chloride	0.000	0.348	Na ₁ Cl ₁
*	98-010-0499	34	Albite high	0.000	0.131	Al ₁ Na ₁ Si ₃ O ₈
*	98-015-9347	31	Orthoclase	0.000	0.074	Al ₁ K ₁ Si ₃ O ₈



Graph 1: Analyze result of raw drug Navachaaram



Graph 2: PSA Analyze result of trial drug Navachaara chunnam

Physico-Chemical Characterization:

Table 3: The Result of Physicochemical Parameters

Parameters	Percentage
Loss on drying	0.64%
Total ash value	97.90%
Acid insoluble ash	33.69%
Water soluble ash	23.12%
Water soluble extraction	32.64%
Alcohol soluble extraction	16.64%
pH	6

Particle Size Analysis:

Table 4: PSA Results of NC

Sample ID	D ₁₀	D ₅₀	D ₉₀
Raw Drug	2.571 µm	15.641 µm	66.532 µm
NC	0.977 µm	5.144 µm	41.707 µm

DISCUSSION

Standardization is a quality control process of assessing the quality and purity of Herbo-mineral drugs, using a wide range of standards, including chemical, biological and physical observation. Standardization of Herbo mineral drugs is significant in order to guarantee the effectiveness,



safety and quality of herbal medicine and integrate it into the current healthcare system. The quality of an end product of a drug is very significant, especially in the pharmaceutical industry. Despite the considerable risk to patients' lives and health, regulatory bodies have taken special care and developed a number of standards to

ensure a suitable level of quality in the pharmaceutical drugs. Navachaara Chunnam is an internal medicine to treat various ailments. Chunnam is one of the thirty-two forms of enteral internal medicine which possesses alkaline properties likely limestone(calcium)⁵ and thus standardization plays a major role to prove its quality.

Table 5: Size and volume distribution of raw drug *Navachaaram*

Size (µm)	Volume %	Cumulate %	Size (µm)	Volume %	Cumulate %	Size (µm)	Volume %	Cumulate %
0.121	0.079	0.079	1.745	1.568	6.911	25.169	5.428	62.343
0.146	0.091	0.169	2.112	1.534	8.445	30.455	5.890	68.233
0.177	0.109	0.279	2.555	1.516	9.961	36.851	6.117	74.350
0.214	0.122	0.400	3.092	1.475	11.436	44.590	5.485	79.835
0.259	0.129	0.529	3.741	2.765	14.202	53.954	5.229	85.064
0.314	0.138	0.667	4.527	4.511	18.713	65.285	4.625	89.689
0.380	0.161	0.829	5.477	5.301	24.014	78.995	3.415	93.104
0.460	0.177	1.006	6.627	4.736	28.749	95.585	2.844	95.949
0.556	0.194	1.199	8.019	4.207	32.956	115.659	2.229	98.178
0.673	0.345	1.544	9.703	4.919	37.875	139.948	1.822	100.000
0.814	0.552	2.096	11.741	4.958	42.833	169.338	0.000	100.000
0.985	0.850	2.946	14.207	5.036	47.869	204.901	0.000	100.000
1.192	1.075	4.021	17.191	4.434	52.303	247.932	0.000	100.000
1.442	1.321	5.342	20.801	4.612	56.915	300.000	0.000	100.000

Table 6: Size and volume distribution of trial drug *Navachaara chunnam*

Size (µm)	Volume %	Cumulate %	Size (µm)	Volume %	Cumulate %	Size (µm)	Volume %	Cumulate %
0.121	0.069	0.069	1.745	4.637	23.611	25.169	2.332	83.147
0.146	0.079	0.148	2.112	3.819	27.430	30.455	2.634	85.781
0.177	0.096	0.244	2.555	3.546	30.977	36.851	2.643	88.424
0.214	0.107	0.351	3.092	2.977	33.954	44.590	2.511	90.935
0.259	0.114	0.465	3.741	4.544	38.498	53.954	2.682	93.617
0.314	0.123	0.588	4.527	6.418	44.915	65.285	2.700	96.318
0.380	0.133	0.721	5.477	7.824	52.739	78.995	1.987	98.305
0.460	0.394	1.115	6.627	7.123	59.863	95.585	1.116	99.420
0.556	0.861	1.976	8.019	5.722	65.584	115.659	0.410	99.831
0.673	1.815	3.791	9.703	4.881	70.465	139.948	0.169	100.000
0.814	2.725	6.516	11.741	3.827	74.293	169.338	0.000	100.000
0.985	3.645	10.161	14.207	2.891	77.184	204.901	0.000	100.000
1.192	4.342	14.504	17.191	1.809	78.993	247.932	0.000	100.000
1.442	4.471	18.975	20.801	1.822	80.814	300.000	0.000	100.000

The Physico-chemical analysis of *Navachaara* chunnam from Table 3 explains the parameters such as Moisture content, Total ash value, Acid insoluble ash, Water soluble ash, Water soluble extraction, Alcohol soluble extraction and pH are within the normal limits. The ingenuity, purity, and quality of the test drug NC are demonstrated by its organoleptic properties, color, texture, taste, and delicate powder nature⁵. The drug NC was brownish in color and it was an odourless fine powdery drug with pH of 6 that reveals it is slightly alkaline and so our stomach will absorb more of it than the intestine⁶. It is apparent that this Herbo - mineral formulation contains significant minerals that maintain the body's normal pH balance and also in simultaneously normalising trace elements uptake in the

body and thus counteracting the disease progression⁵. The loss on drying value represents the drug's moisture content, which was assessed as 0.64%. The moisture content of the Herbo mineral drug should be minimal since it promotes the growth of live organisms, fungi, or insects and causes degradation on subsequent hydrolysis⁵. By this way the study helps to standardise the preparing procedure of this Herbo mineral composition.

The total ash value of the test drug NC was 97.90% and the value of insoluble acid ash was 33.69% showing the purity of the trial drug. One of the most important quantifiable elements for the herbo mineral drug's standardization is its ash value. An elevated ash value indicates a higher

concentration of inorganic elements⁶. The water and alcohol soluble extractive values were found as 32.64 % and 16.64% respectively. Extractive values are helpful in determining the percentage of chemical soluble in a specific solvent and in determining the amount of phytoconstituents present in the herbo mineral medicine⁵. The trial drug NC has a pH value of 6 indicates that it is weekly alkaline. A drug's pharmacological activity and bioavailability are influenced by its solubility property. The trial drug NC is highly soluble in water and soluble in ethanol, insoluble in chloroform and ethyl acetate.

The chemical composition of the trial drug NC can be determined by XRD. Pure substances have unique X-ray diffraction patterns, similar to fingerprints¹⁰. The powder diffraction method is ideal for characterising and identifying polycrystalline phases. Currently, over 50,000 inorganic and 25,000 organic single components, crystalline phases, and diffraction patterns are preserved on magnetic or optical media as standards¹⁰. Powder diffraction is primarily used to identify sample components using a search/match method¹⁰.

The full peak list includes the position, height, Full Width at Half Maximum (FWHM), d-spacing, and relative intensity of each detected peak. Notably the XRD pattern of the test drug NC reveals the predominant presence of quartz as evidenced by the prominent and sharp peaks at specified 2θ values at 26.5877° (37882.26 cts). The Other prominent peaks include 20.8174°, 27.9582° and 31.6142°. The particles of NC shows the crystalline composition nature. The presence of sodium chloride, albite, and orthoclase in the sample indicates a complex mineral composition, potentially contributing to its therapeutic qualities. The prominent evidence of presence of Quartz was revealed from the sharp peaks at 2θ values at 26.5877° (37882.26 cts). Further from this observation it was concluded that Quartz and sodium chloride may be the key ingredients present in the test sample NC. The presence Of Quartz which is required for wound healing property found in Navachaaram may aids to amplify healing energy and is particularly effective for chronic fatigue, arthritis, bone injuries, depression, diabetes, fibromyalgia and intestinal troubles. It contains Manganese, a trace essential mineral for human health.

The particle size dimension of the trial drug NC was sized up to be 41.707 μm which was comparatively smaller than the particle size of raw drug Navachaaram which was found to be 66.532 μm. It also implies that Fifty percent of the raw material falls under the particle size of 15.64 μm. Similarly fifty percent of the trial drug falls under 5.144 μm. In this way, it is obvious that the size of the particle is capable to encourage the Efficacy and it shows the major transformation of the drug to the finished product.

CONCLUSION

This study reveals the purity and bioavailability of the Navachaara chunnam. The analysis expounds the presence of essential trace elements in test drug NC which is

necessary for a variety of vital biological activities. Moreover, pharmacological research must be carried out to validate the therapeutic value of Navachaara chunnam.

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