

Seperation Of Impurities From Talc Ore By Flotation And Dissolution

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Abstract:

Talc is a main raw material for talcum powder and an important ingredient in ceramics, paint, paper, plastics, rubber and insecticides. Talc ore contaminated with calcite mineral was beneficiated by flotation and dissolution. The option of flotation was explored because talc is sufficiently hydrophobic and easily floatable compared to other gangue minerals. Only frothing agents such as poly ethylene glycol (PEG), pine oil and methyl isobutyl carbinol (MIBC) were used to stabilize the froth. Though the talc was enriched in froth product, complete elimination of calcite could not be achieved by flotation. The CaO content in talc was brought down to just 11.5% from its initial value of 15% by flotation. Hence, dissolution of calcite using HCl, acetic acid, formic acid, and oxalic acid was attempted. The elimination of calcite by dissolution was found to be effective in the presence of acids. Though the HCl was found to be effective, the brightness of talc was found to decrease. Among the organic acids attempted, acetic and formic acids were found to be effective and advantageous because the byproducts are easily soluble in water and can be separated by filtration. The reaction between calcite and acetic/formic acid was found to proceed in the mole ratio of 1:2 respectively.

Key words: Talc, calcite, flotation, separation, dissolution, organic acids

INTRODUCTION

Talc is an industrial mineral that belongs to the family of phyllosilicate. It is a main raw material for talcum powder because of its ability to absorb moisture and oils. Talc is also an important ingredient in ceramics, paint, paper, plastics, rubber and insecticides. Talc ores are generally associated with sheet silicates such as serpentine, dolomite $[(Ca,Mg)CO_3]$, magnesite $[MgCO_3]$, quartz $[SiO_2]$, Breunnerite $[(Mg,Fe)CO_3]$, calcite $[CaCO_3]$, siderite $[FeCO_3]$, ankerite $[Ca(Fe,Mg,Mn)(CO_3)_2]$, feldspar $[(K,Na)AlSi_3O_8]$, magnetite $[Fe_3O_4]$, rutile $[TiO_2]$, Pyrite $[FeS_2]$, pyroxenes, olivine, biotite and amphiboles [1]. In certain places of Egypt, talc is present as bands or lenses in the plagioclase-quartz-biotite schist.

Since talc is naturally hydrophobic, it is separated from gangue minerals by flotation technique. Instead of conventional ball and rod milling, attrition technique was followed as pre concentration [2]. The talc chlorite was improved to 82% by attrition in the presence of sodium hexametaphosphate at a pulp density of 76% [3]. Beneficiation of talc-carbonate ore using polypropylene glycol as a frother was studied to increase the rate of talc flotation [4]. A clean concentrate analyzing 93.5 % talc was obtained at a recovery of 70% from an ore containing 48.69 %. The effect of various frothers on flotation rate, and entrainment of fine calcite particles were investigated using individual minerals and a mixture of talc and calcite [5]. The results indicated that the type of frother had significant effect on the flotation rate and entrainment. The selectivity between talc and calcite was enhanced by the addition of kerosene as a collector. This was attributed to increased flotation rate of talc without enhancing the recovery of calcite fines. In yet another study, the effect of phytic acid and sodium dodecyl benzene sulfonate on talc separation from the ore contaminated with asbestos was attempted [6]. Concentrate assaying 61.87% of SiO_2 and 0.60% of CaO was achieved by adopting a flow sheet consisting of roughing, three stage cleaning and scavenging. The adsorption of guar gum and dextrin on talc was studied and found that guar gum exhibit better depressant ability

compared to dextrin [7]. The flotation behavior of talc products having different particle shapes produced by different grinding mills (ball and rod mill) was evaluated using column flotation process [8]. It was observed that the particles of higher elongation and flatness were recovered better during column flotation, whilst spherical and relative width had a negative effect on the flotation behavior of the talc. The ultrasonication of talc was found to improve the depressant action of guar gum (IMP4) and carboxymethyl cellulose (CMC). The recovery of talc was decreased by nearly 30% after 4 minutes of ultrasonic pre conditioning [9]. The micro topographic studies by atomic force microscopy indicated that surface defects formed by ultrasound could serve as active sites for water adsorption rendering the naturally hydrophobic talc to hydrophilic.

In the present investigation, different frothers and depressants were tried to upgrade the talc containing free silica and calcium carbonate. Different acids were also tried to remove calcite by dissolution process to improve the overall quality of talc.

2. EXPERIMENTAL MATERIALS AND METHODS

2.1 Materials:

Talc ore and pure talc lumps were obtained from the mines situated in Ananthapur, Andhra Pradesh, India. Frothers namely poly ethylene glycol (PEG), pine oil and methyl isobutyl carbinol (MIBC) were procured from Alfa Aesar. All the other chemicals used are of analytical grade. The reagent solutions were prepared in double distilled water.

2.2 Chemical analysis:

The chemical composition of various fractions of flotation was carried out by standard procedure [10].

2.2.1: Loss on ignition: The sample was weighed in a platinum crucible and placed in a muffle furnace. The temperature of the furnace was slowly raised to 950 - 1000°C. After about 30 minutes, the crucible was removed and cooled in a desiccator and its weight was

recorded again. The ignition and cooling was repeated till the constant mass is obtained. From the difference in weight, the percentage of loss was calculated.

2.2.2: Determination of silica: The sample was decomposed by fusion with sodium carbonate and extracted with hydrochloric acid. Silica was then determined gravimetrically by dehydration and baking followed by hydrofluorization. The residual silica in the filtrate was determined photo metrically using molybdenum blue method.

2.2.3: Determination of CaO and MgO: In an aliquot of the sample solution, R_2O_3 group elements were masked by triethanolamine, and calcium and magnesium were titrated with EDTA solution at pH 10 using eriochrome black T indicator. In another aliquot magnesium was precipitated out at pH 12 in the presence of triethanolamine and calcium is titrated with EDTA using Patton and Reader (P & R) indicator. The titration value for magnesium was obtained by subtracting the titration value of calcium from that of calcium and magnesium. From the complexometric titration, the CaO and MgO were determined.

2.3 BET Surface area:

The specific surface area and porosity was determined by BET analyzer (Brunauer, Emmett and Teller) Model ASAP 2020 supplied by Micromeritics, USA.

2.4 Contact angle measurement:

Contact angle of the mineral powders was determined by sorption measurement using Kruss Tensiometer, model- K 100. The contact angle of the powder sample was arrived from the Washburn equation incorporated in the software. When a column of powder bed is in contact with liquid, the pores between the particles act like small capillaries and the rise of liquid is measurable. The capillary constant in the Washburn equation was determined using n-hexane. The mineral sample was evenly packed in the sample tube each time. Measurement was repeated at least 3 times and an average value was reported.

2.5 Surface characterization:

Scanning probe microscope, model 5500 series, Agilent Technologies, USA was used for surface characterization. Measurements were made in contact mode using NSC 15 Cantilever and Picoview 1.12.2 software. Silica wafer samples were cut to size of the sample holder and immersed in the aqueous solutions and subjected to ultrasonication treatment for 30 minutes to remove the adhered slimes. The fresh sample was equilibrated with amine solution for 30 minutes and dried in vacuum desiccators. The dried samples were examined by scanning probe microscope.

2.6 Flotation tests:

Batch scale flotation tests were conducted in D-12 Denver flotation machine. The flotation tests were conducted at 25% solids at an impeller speed of 1200 rpm. The pH was adjusted prior to the addition of depressant. The conditioning time of 3 minutes was maintained for each reagent. The floated fraction and residue was collected separately and analyzed.

3.0 RESULTS AND DISCUSSION:

3.1 Characterization: The mineral phases present in talc sample was identified using X-ray powder diffraction. The diffraction pattern was indexed using inter planar spacing (d-value), peak position (2θ degree) and peak intensity. The mineral phases were indexed by matching with the powder diffraction standard database maintained by International Center Diffraction Data (ICDD). The XRD data reveals the presence of calcite, talc and quartz mineral phases in talc.

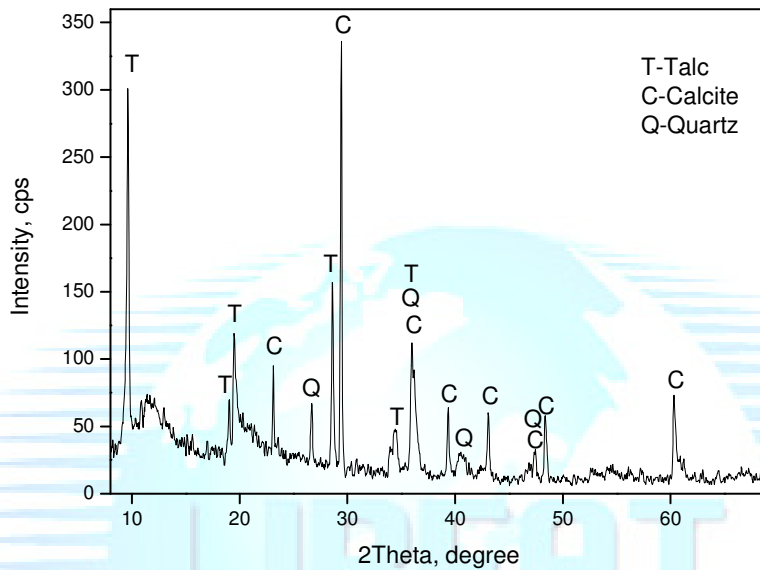


Fig 1: X-ray diffractogram of talc sample

In order to avoid the contamination, the talc sample was ground in a pulverizer equipped with agate jars and balls. The microscopic data has revealed that the talc was liberated from calcite below 45 microns. The particle size distribution ground sample was carried out using CILAS particle size analyzer and the data was shown in Fig 2. It is evident that the mean diameter of particles is 21.47 microns and 10% of the particles are less than 2.39 microns. During grinding or milling two types of surfaces are expected. One type of surface result from the easy breaking of the Van der Waals bond of the surface from its neighbour which forms a basal face that is hydrophobic. The other arises due to rupture of ionic/covalent bonds within the layers whose edges are hydrophilic in nature. It was reported that 90% of talc is hydrophobic due to the siloxane surface with the inert –Si-O-Si- links that are non polar. The contact angle measurements conducted on talc ore indicated high degree of hydrophobicity with contact angle of 80.57 degrees. Hence there

is no need to add the reagent to make the talc surface hydrophobic. Therefore flotation experiments were carried out using just frother alone.

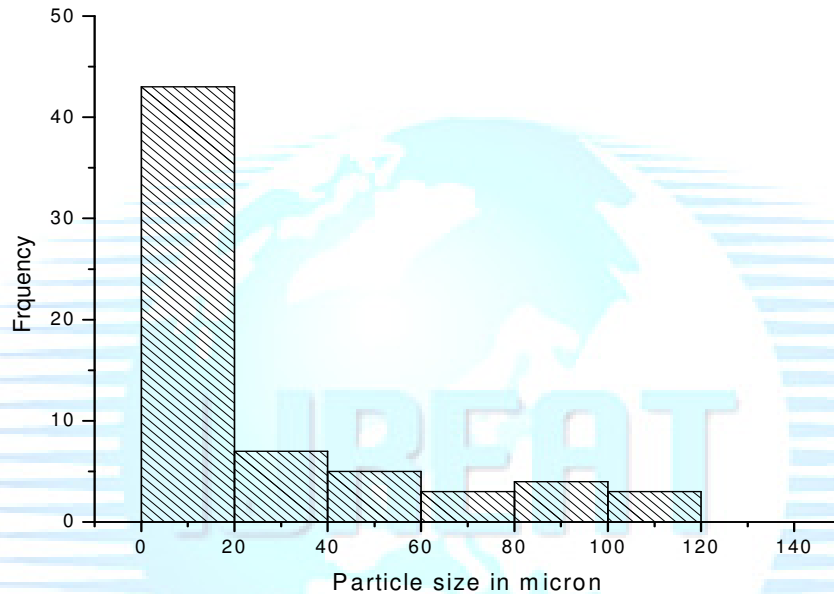


Fig 2: Particle size distribution of talc sample

Effect of frother and its concentration:

The effectiveness of different frothers viz. poly ethylene glycol (PEG), pine oil and methyl isobutyl carbinol (MIBC) was studied and the results are presented in table 1. In general, molecular weight, hydrophile-lipophile balance, dynamic frothability index and critical coalescence concentration are important parameters in determining the effectiveness. For example higher molecular weight frother results in more persistent froth and less selectivity than lower molecular weight frothers [11]. The results indicate that MIBC is more effective and selective compared to pine oil and PEG. The MgO content in talc was improved to 25.5% and CaO was decreased to 12% from its original

value of 15.5 %. The recovery of talc is also better in the presence of MIBC compared to other frothers. It was reported that glycol type frothers are preferred wherever fine bubbles are required. MIBC and pine oil are used to generate medium and fine bubbles respectively [12]-[13]. The effectiveness of MIBC may be attributed to the optimum ratio of particle to bubble size that is suitable for better collision.

Frother	Dosage (kg/t)	Weight (%)		Float assay (%)		Sink assay (%)		Recovery (%) MgO
		Float	sink	MgO	CaO	MgO	CaO	
PEG	0.137	38.46	61.54	24.34	12.32	22.75	17.49	38.73
PEG	0.183	42.07	57.93	25.09	13.38	20.46	17.04	49.24
Pine oil	0.0764	50.50	49.50	24.44	12.82	22.26	18.24	52.84
Pine oil	0.1146	52.00	48.00	24.80	13.63	21.80	17.53	55.21
Pine oil	0.1528	66.27	33.73	25.17	15.33	22.44	15.83	63.65
MIBC	0.079	70.12	29.88	22.64	14.22	25.05	18.50	67.96
MIBC	0.119	68.21	31.79	25.50	12.00	18.77	23.01	74.46
MIBC	0.159	79.63	20.37	28.09	10.42	22.15	16.80	75.51

Table 1: Effect of frother and its concentration on grade and recovery of talc

Conditions: Particle size (d_{80}): 36 μ m, Sodium hexameta phosphate: 2.0 kg/t, Slurry pH: 7.0, Conditioning time: 3 minutes.

Effect of slurry pH:

The effect of slurry pH on grade and recovery of talc was also studied and the results were shown in Table 2. It is evident that the quality of talc is better at pH 10.

Frother dosage (kg/t)	pH	Weight (%)		Float Assay (%)		Sink Assay (%)		MgO recovery of (%)
		Float	Sink	MgO	CaO	MgO	CaO	
Pine oil (0.1528)	10.0	55.89	44.11	26.03	11.45	19.98	20.63	62.28
Pine oil (0.1528)	9.0	54.77	45.33	24.90	11.36	21.50	20.51	58.37
MIBC (0.0792)	10.0	45.55	54.45	24.82	11.41	22.14	18.92	48.39
MIBC (0.0792)	9.0	51.33	48.67	25.32	11.67	21.29	19.54	55.65

Table 2: Effect of slurry pH on grade and recovery of talc

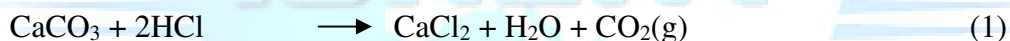
Conditions: Particle size (d_{80}): 36 microns, Sodium hexameta phosphate: 2 kg/t,

Conditioning time: 3.0 minutes

From the earlier literature, it is known that the iso electric point of talc, quartz and calcite are 1.7, 2.9 and 8.5 respectively. By adjusting the pH to above 9.0, the charge of these minerals will become negative and thus all the minerals are dispersed effectively by electrostatic repulsion. Though the quality of talc concentrate was slightly improved, the CaO content remains high even at pH 10. Experiments on natural floatability of talc (without reagents), split addition of reagents, combination of oleic acid, diesel oil and MIBC was attempted to eliminate calcium carbonate. Starch also was tried as depressant towards calcium carbonate. However the desired results could not be achieved. The reason for poor selectivity may be interpreted due to the presence of large quantity of slimes (10%) generated during grinding. The poor floatability of fines in many circuits is generally attributed to poor collision probability. The fines require high collector dosage and more flotation time. The fines and coarse particles will never float equally in the same cell, because it is difficult to optimize the reagents for both. Surface analysis of fines lost to tailings invariably shows that they are not hydrophobic enough. Adding extra

collector will float the latter particles, but at the cost of floating gangue minerals [14]. In conventional flotation cells, the bubble size is sufficiently large compared to the fines and thus the collision probability is low. The fine particles of talc tend to adsorb on coarse particles of calcite and makes the calcite particles to float. Thus the quality of the talc is seriously affected due to fines.

Since the talc quality could not be improved by flotation, and the major contaminant being calcite, dissolution of calcite by dilute acid was opted. The 100 g talc sample was treated with each acid and filtered through whatman filter paper and washed thoroughly with double distilled water. The precipitate was dried separately and chemical assay of talc concentrate was conducted and the results are presented in Table 3. The dissolution of calcium carbonate with dilute HCl, acetic acid, formic acid, succinic acid, oxalic and citric acids was attempted. The chemical reaction involved between CaCO_3 and HCl is given in equations 1.



From the results presented in Table 3 and 4, it is evident that carbonate can be effectively removed in all sieve fractions and dilutions studied. The acid consumption was found to increase as the particle size is decreased. Though the dissolution of CaCO_3 by HCl is most effective and cheaper, it is not considered because of corrosive nature and presence of iron oxide in talc. The iron oxide is dissolved as FeCl_3 and affects the brightness of talc. Further, the adsorption of chloride ion on talc at neutral pH is high and hence deleterious for skin application. Above all, it is difficult to prevent the equipment from chloride corrosion.

Table 3: Effect of particle size on calcite dissolution

Initial assay of talc: SiO_2 : 42.75 (%), CaO : 15.5 (%), MgO : 19.53 (%), CO_2 : 15.57%

Concentration of HCl: 2.828 N

Sieve fraction	+150 μ	-150 + 106 μ	-106 + 75	-75 μ
MgO	30.38	30.30	30.35	30.18
CaO	3.32	3.64	3.58	3.63
SiO ₂	60.46	60.18	60.01	60.10
Acid consumption	120.5 ml	132 ml	155 ml	157 ml

Table 4: Effect acid/water ratio

Initial assay of talc: SiO₂: 42.75 (%), CaO: 15.5 (%), MgO: 19.53 (%), CO₂: 15.57%

HCl: water ratio	1:10	1:15	1:30
MgO	30.38	30.19	30.55
CaO	3.22	2.80	3.17
SiO ₂	60.27	60.48	59.72
Acid consumption	233 ml	345 ml	460 ml

Selective dissolution of CaCO₃ in aqueous solution of weak organic acids such as acetic acid, formic acid, succinic acid, oxalic and citric acid was attempted and the results are presented in Table 5. In the presence of acetic and formic acid, the calcite was totally dissolved as per the reactions mentioned in equations 2 and 3.



As a result, the MgO content of talc was improved to above 31% and SiO₂ to 61%. While the carbon dioxide escapes to atmosphere, the calcium acetate and calcium formate which are soluble in water can be filtered and separated from talc. The solubility of calcium

acetate and calcium formate in water is 34.7 g/100 ml and 16.6 g/100 ml respectively. Dissolution of calcium carbonate in aqueous solutions of acetic acid is found to be a diffusion controlled process with an apparent activation energy of 17.2 kJ. mol⁻¹ [15]. The diffusion control of the process was attributed due to formation of a gaseous CO₂ film on the CaCO₃ surface. The order of the CaCO₃ dissolution reaction with respect to acetic acid is 0.5. The reactivity of formic acid on calcium carbonate particles was studied [16]. Along with calcium formate, adsorbed carbonic acid was also identified on calcite surface. The adsorbed layer was found to inhibit the additional uptake of formic acid. However, the adsorbed carbonic acid which is highly unstable, dissociates into CO₂ and H₂O in the presence of water. The surface reaction can be represented by the following reaction.

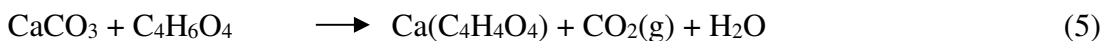


In the present study, two moles of formic acid/acetic acid was consumed for the complete dissolution of 1 mole of calcium carbonate. Thus it is apparent that water soluble complexes of calcium acetate and calcium formates are formed during the dissolution process.

Though the calcium carbonate is dissolved in the presence of succinic acid and citric acid, the solubility of their respective calcium complexes in water is poor (calcium succinate: 0.2 g/ 100 ml, calcium citrate: 0.095 mg / 100 ml). Consequently the calcium complexes of succinic acid and citric acid remain with talc as separate solid phases. Though these by-products can be separated using suitable solvents, the process becomes cumbersome and its economic viability needs to be evaluated. Though the dissolving ability of organic acids is relatively weak, they exhibit high selectivity towards leaching of calcareous particles from talc. On the other hand, high CO₂ pressure and froth formation was observed due to faster dissolution while using HCl. Thus the organic acids may be promising extracting agents as the leaching is carried out at intermediate acidic conditions (pH:3.0-5.0) and the degradation of these acids is biologically amenable.

The reaction between succinic acid and calcium carbonate is shown in equation 5.

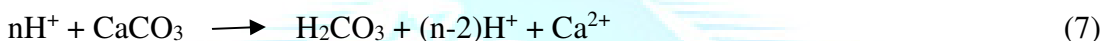
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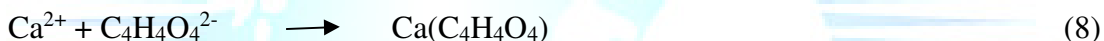
This reaction may also include the formation of unstable carbonic acid which in turn decomposes to CO_2 and H_2O and also ionization of succinic acid as shown in equation 6.



The H^+ ions thus generated reaches the calcite surface by diffusion process and attack calcium as per the following mechanism.



The Ca^{2+} ions thus dislodged from the surface in turn reacts with succinate ion and form calcium succinate complex as per the equation 8 shown below.



The details of various reactions describing the leaching of calcium ions and complexing of Ca^{2+} with succinic acid are described elsewhere [17].

Quantity of acid added	Weight (%)	SiO_2 (%)	CaO (%)	MgO (%)	Fe_2O_3 (%)
Acetic acid 20 ml	83	53.46	11.0	20.14	1.92
Acetic acid 30 ml	73	61.5	1.5	31.85	1.65
Acetic acid 40 ml	72	61.8	0.11	32.0	1.24
Formic Acid 10.ml	87	49.98	12.52	19.92	1.88
Formic Acid 20 ml	73	60.5	2.05	31.25	1.72
Formic Acid 30.ml	74	61.78	1.6	29.41	1.02
Succinic acid	>100 g	46.88	13.1	19.5	1.88
Oxalic Acid	>100 g	44.77	12.5	20.5	1.95
Citric acid	>100 g	45.77	13.56	18.66	1.74

Similarly, the calcite was found to dissolve in the presence of citric acid. Since the solubility of calcium citrate in water is 0.095 g/100 ml, it forms insoluble residue along with talc. Citric acid easily ionizes in water to form citrate ions viz H_2AOH^- , HAOH^{2-} , AOH^{3-} and AO^{4-} (Where A is $\text{C}_6\text{H}_4\text{O}_6$). The H^+ ions released during ionization of citric acid initiate the dissolution of calcite. The Ca^{2+} thus formed in turn reacts with citrate molecules to form calcium citrate complex.



Based on equation 11, it is evident that the precipitation of calcium citrate is dependent on pH value and can occur at or above pH 6.0. The reaction of citric acid with calcium was examined using rotating disc electrode and concluded that the reaction rate was limited by the precipitation of calcium citrate on calcite surface [18].

FT-IR Study:

The FT-IR Spectrum of talc ore after acetic acid treatment was recorded and shown in Fig 3. Two prominent peaks at 1060cm^{-1} and 665cm^{-1} are characteristic to Si-O stretching vibrations [19]. The stretching vibrations of metal oxide (M-O) are generally observed below 450cm^{-1} . However in the case of SiO_2 , the Si-O vibrations could be seen around 1060cm^{-1} . The sharp peak around 3600cm^{-1} may be attributed to stretching vibrations of O-H adsorbed on talc surface. Numerous narrow peaks observed between $4000-3500\text{cm}^{-1}$ may be due to adsorbed water molecules on talc surface. It may be noted that the characteristic bands of CaCO_3 around 1785cm^{-1} and 877cm^{-1} are absent in the spectrum. Thus it is apparent that the calcite was dissolved in acetic acid.

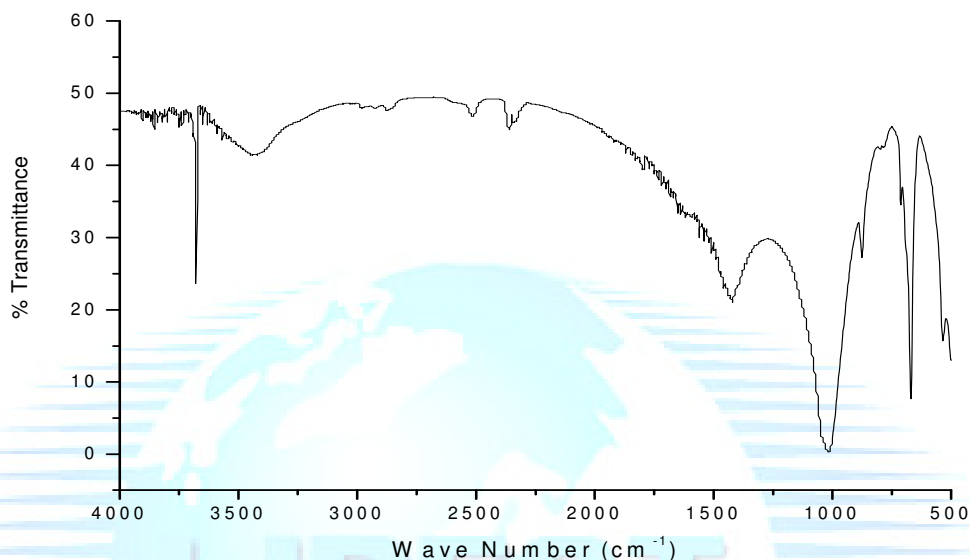


Fig 3: FT-IR Spectrum of talc ore after acetic acid treatment

The doublet at 2340-2360 cm⁻¹ is assigned to CO₂ adsorbed on talc during dissolution.

The FT-IR spectrum of filtrate shown in Fig 4 exhibited strong bands in the region of 1340-1600 cm⁻¹. The bands around 1420 cm⁻¹ and 1554 cm⁻¹ are assigned to symmetric and asymmetric vibrations of COO⁻ ion respectively [20].

PRDG

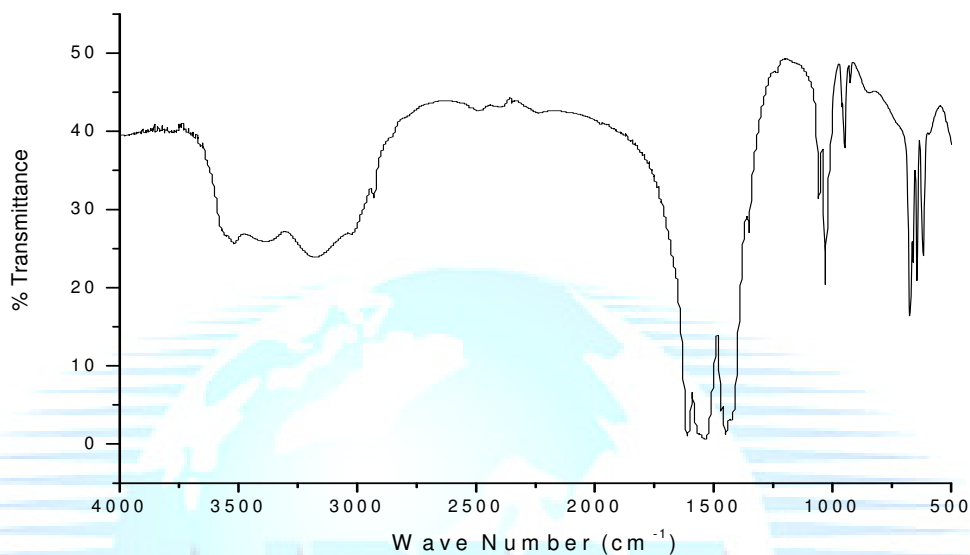


Fig 4: FT-IR spectrum of filtrate resulted from acetic acid treatment of talc.

The absence of sharp band around 3600 cm^{-1} indicates the absence of O-H group. Similarly, the band at 1712 cm^{-1} assigned to C=O stretching is also absent in the spectrum. This clearly indicates that the acetic acid is in the form of acetate ion bonded to calcium ion dissolved from calcite. The broad bands in the region of $3100\text{--}3600\text{ cm}^{-1}$ are attributed due to hydrogen bonded O-H groups.

The FT-IR spectrum of talc ore treated with formic acid and the filtrate solution are presented in Figs 5 and 6 respectively. The spectra are more or less similar to the spectrum of talc ore treated with acetic acid. The formate is characterized by the bands at 1350 and 1580 cm^{-1} usually assigned to symmetric and asymmetric vibrations of COO^- respectively [20] [21]. The sharp band at 3600 cm^{-1} and the broad band around 3400 cm^{-1} are explained due to free surface hydroxyl groups and hydrogen bonded hydroxyl groups.

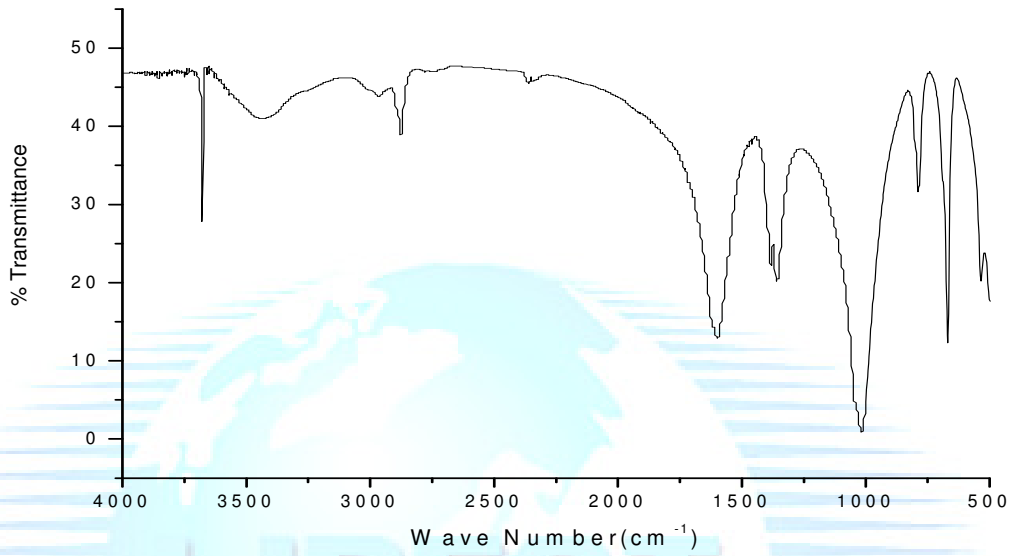


Fig 5: FT-IR Spectrum of talc ore treated with formic acid

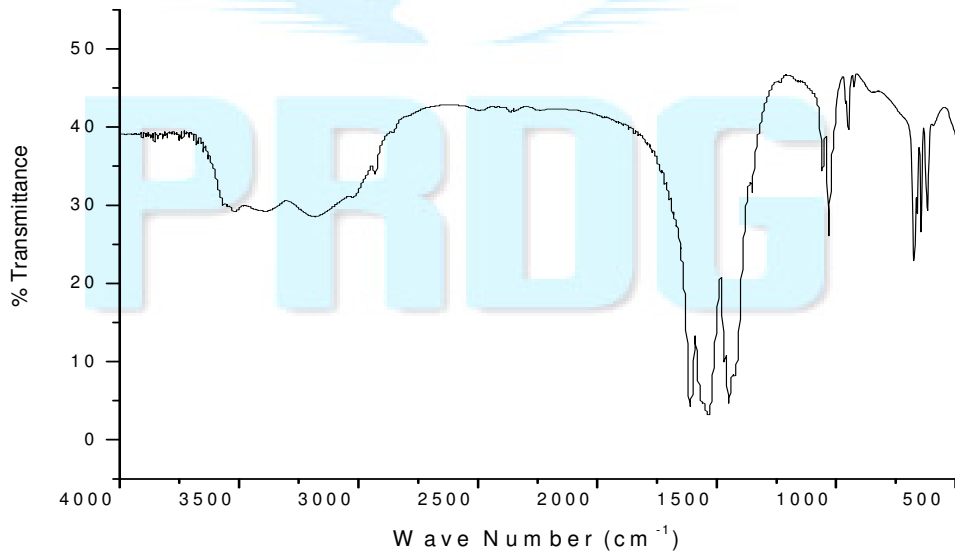


Fig 6: FT-IR spectrum of filtrate resulted from formic acid treatment of talc.

The characteristics of spectra of filtrate solutions of acetic and formic acids shown in figures 4 and 6 are similar. Hence it could be concluded that calcium formate is formed as byproduct. Because of high solubility of calcium acetate and calcium formate in water, these two products can be separated from talc ore simply by filtration. Thus calcite can be easily dissolved in the presence of acetic and formic acids and the quality of talc can be improved. The dissolution of calcite was attempted using oxalic, succinic and citric acids. Though the calcite was dissolved in the presence of these acids, it is difficult to separate the resultant products namely calcium oxalate, calcium succinate and calcium citrate from talc due to poor solubility in water. However these byproducts can be separated from talc using suitable solvents.

Summary and Conclusions:

Beneficiation of talc by flotation and dissolution was studied and the following conclusions were drawn.

1. Separation of talc from calcite gangue by flotation using poly ethylene glycol (PEG), pine oil and methyl iso butyl carbinol (MIBC) as frothers was investigated. Perfect elimination of calcite from talc could not be achieved by flotation.
2. The dissolution of calcite was found to be very effective in the presence of HCl. But the brightness of talc was decreased.
3. The dissolution of calcite in the presence of weak organic acids such as acetic, formic and oxalic acids was found to be complete. Among these acids, acetic acid and formic acid are advantageous because the byproducts can be separated by simple filtration process. Solvent extraction process is essential in the case of citric acid and oxalic acid to separate the byproducts.

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