

## Literature Review on the Methodology

### 2.1 BACKGROUND

Apart from allaying wind and seismic damages, mud wall serves as a natural conditioner of air [Blondet and Garcia, 2004; Houben and Guillaud, 1994; Satankar *et al.*, 2018]. Small houses can have resistance to natural fractures due to movements of earth, since they are strengthened by concrete columns and structurally reliable beams [Blondet and Garcia, 2004]. Improvement of mud-based structures may be necessary to reduce such damages [Blondet and Garcia, 2004]. Ecological and aesthetic benefits of mud construction attract the attention of government agencies, researchers, intellectuals, contemporary architects, eco-builders [Satankar *et al.*, 2018]. Mud construction leads to an indoor environment beneficial to human health [Satankar *et al.*, 2018]. Recent trends of nature (eco) tourism derive a lot of green dream spaces using these traditional mud-based materials to cater India's soft power to international visitors [Ramkumar, 2017]. Unbaked mud and other related natural organic materials reduce energy consumption [Keefe, 2005]. Mud-based housing has low embodied energy and low carbon dioxide emissions [Venkatarama, 2009]. The delusion associated with mud-based construction is the assumption that mud is only used for housing in poor rural areas but there are examples (like Auroville, Tamilnadu) of industrial and urban infrastructure that are made of the earth [Maini, 2010; Venkatarama, 2009]. Construction of local materials can also benefit the local economy, embalm environment and restore traditions [Ejiga *et al.*, 2012; Kidman, 2017].

The present contemporary manufacturing and residential spaces are generating solid wastes and creating open waste dumps in urban India's neighbourhoods [Pappu *et al.*, 2007]. This basically creates another dimension of importance to the current work since animal dung, carpentry scrap is categorized under wastes. Efficient utilization of some of these materials had been embedded within the traditional devices which are being used in Jodhpur, Rajasthan [Kaurwar *et al.*, 2017; Pappu *et al.*, 2007; Satankar *et al.*, 2017]. This also asserts that rural waste material utilization techniques are technologically sound thus accentuates investigation and to be elaborated in a scientific parlance rather than ethnography. Moisture attacks mud, reducing its elastic modulus and shear strength (due to decrease in cohesion among soil particle) and enhances wear (erosion by rain) with adequate contact time with water [Saxton and Coventry, 1995; Mouazen *et al.*, 2002]. This technological concern can be solved by additives such as animal dung and other organic materials [Agarwal, 1981]. Recently corn cob powder-based aggregates are being used for producing lightweight concrete masonry structures [Faustino *et al.*, 2015].

But as early as 1981, UNCHS supported a research predicting and reiterating mud as a potential housing construction material for developing nations like India [Agarwal, 1981]. Further, expensive and energy-intensive construction materials have been substituted or partially replaced with recycled industrial, agricultural, human waste, organic bio-based materials and plastics [Azeko *et al.*, 2015; Azeko *et al.*, 2015; Gupta, 2014; Mustapha *et al.*, 2015; Savastano *et al.*, 2000; Della, 1988]. This posits a closed cycle of environmentally safe constructional material alternatives which can reduce energy, labor and cost [Milani, 2005]. The use of green material for constructions might have been considered stoked in the sixties in the urban west but in India, these materials have been sustainable from the Vedic periods [Milani, 2005; Dikshit, 1974; Iyer, 1884]. In this thesis, such material structures with its provenance in arid areas near to the Thar desert is depicted.

### 2.2 SOIL-BASED TECHNOLOGIES IN LOCAL COMMUNITIES

#### 2.2.1 Materials from Western Rajasthan Used in Construction

Adobe is the oldest known affordable material in building construction [Minke, 2012, Wolfskill *et al.*, 1963]. It is basically soil stabilized by adding a straw, dung and water [Bahobail, 2012]. Soil stabilization using local materials found in the arid areas of western Rajasthan has been performed. For example, one of such species is *Crotalaria Burhia* (CB) has found its application as a soil binder along with its use as fodder for cattle under extreme conditions for ages in Marwar region [Dave *et al.*, 2017]. Here, on addition to soil

it not only provides strength to the mud-based structures these materials also said to provide cooling effect when used as a construction material [Dave *et al.*, 2017]. Local climate and material availability strongly influence rural construction material use [Brunhes, 1920]. The use of these materials also reflects on the location's heritage, ecology (flora and fauna) as well as origin of settlements and their traits [Dickens and Pitts, 1963]. Similarly, Neem and Bamboo are locally available and used in this *Mewar* region to provide truss or gusset plate or spacer block elements respectively [Shrimali *et al.*, 2014]. *Khimp* is the plant material locally used for making the roofing of circular (wind and earthquake protection) shaped soil-dung-straw stabilized wall houses in western Rajasthan [Dave *et al.*, 2017, Satankar *et al.*, 2017].

Goat dung and its hair are also used in this region for soil stabilization and manufacturing flywheels for pottery use respectively [Roux, 2015]. Camel dung contains small fibers which make them preferable to be used as additives in Rajasthan because it makes the arid soil workable for plastering and rendering [Ilse Köhler-Rollefson, 2004]. Cow dung is basically used in India for mud floor finishing and disinfection of permeable flooring [Girija *et al.*, 2013; Roger, 1902]. It is also used to soil stabilizer and as a plaster for pottery kilns [Wolfskill *et al.*, 1963; Roux, 2015]. Cow dung used for biogas production, once digested beyond gas production is dried and compressed to form fiber composite stronger than their wooden ply counterparts. The traditional belief and thought process might be the responsible reason behind the sustainable use of dung in different process of daily use [Satankar *et al.*, 2017].

**Table 2.1:** Housing scenario at Rajasthan (based on the material used in wall construction) [Source: Census of India, 2011]

Material for construction	Total	Rural	Urban
Organic Plant Material	4,24,345	4,03,385	20,960
Plastics	24,471	18,405	6,066
Mud/ Unbaked brick	32,69,015	30,89,906	1,79,109
Wood	83,283	70,605	12,678
Stones (unpacked with mortar)	11,25,928	8,75,259	2,50,669
Stones (packed with mortar)	62,26,520	47,75,445	14,51,075
Metal/ Asbestos sheets/Galvanized	25,769	14,789	10,980
Baked brick	48,41,930	27,46,728	20,95,202
Cement Concrete	64,370	26,657	37,713
Others	67,203	42,653	24,550
<b>Total</b>	<b>161,52,834</b>	<b>120,63,832</b>	<b>40,89,002</b>

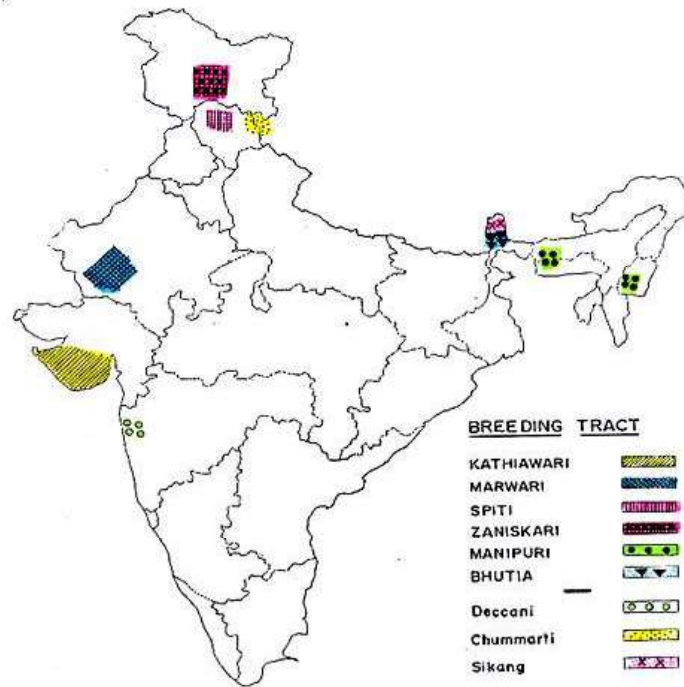
Apart from these organic materials, several other materials used for housing construction in rural areas of Rajasthan are listed in Table 2.1. According to 2011 census (illustrated in Table 2.1) in Rajasthan state 25.6% are adobe houses and 7.2% of mined stone (with no mortar) houses. Additionally, since the region is dotted with numerous stone quarrying sites and mining of marble, granite, sandstone, limestone continue unabated for centuries. Stones are traditionally piled up without any adhesive or mortar support by traditionally skilled labor to erect wall supports for a *Khimp* roofed house. Several such houses are constructed in Marwar region due to mined waste stone availability at negligible costs.

#### (a) Horse Dung for Construction

From Figure 2.1 the population of horses in Rajasthan is clearly shown to be limited to the Marwar region. The technology related to the use of equine ordure also is found within these limits. This natural habitat is an arid desert. The locals of the region due to its harsh weather were prompted to use materials found within their close vicinity. For centuries people of this region managed to survive the environment using material properties of Equus waste. In this regard, another positive dimension is the traditional use of Equus waste as a medicine which cures bacterial infection such as common colds cough etc. This may be a possible reason for using equine ordure as an element for building construction.

A depletion of this natural material is connected to the slowly fading Equus population [NRCE, 2015]. Further, Equus in this region are rapidly deteriorating in their quality as a result of lack of organized breeding and availability of good (specimen) animals. Therefore, the sustainability and propagation of this

construction material technology may require support of Equus species conservation. The equine ordure is traditionally said to good for structural applications due to its visibly large fibrous content as compared to cow dung.



**Figure 2.1:** Distribution of equine population in India [Source: NRCE, 2015]

Recently, a team of researchers at ETH Zurich and the University of Bonn led by Markus Aebi, professor of Mycology tried to understand how horse dung grown fungus and other microbes affect each other during their growth within close vicinity. This involved growing the fungus along with other microbes in a closed compartment and performed tests to study their coexistence with each other. They discovered *Coprinopsis Cinerea* that grows on Equine ordure is able to kill specific microbes [Essig *et al.*, 2014]. Further, they demonstrated that the copsin produced by *Coprinopsis Cinerea* is having antibiotic effects. Basically, Copsin is defensin, which are small proteins produced by certain organisms to combat microbes or bacteria that may cause diseases [Essig *et al.*, 2014]. Copsin can kill many pathogens that can cause severe food poisoning. This research supports the thought of Equus dung as a traditional medicine against bacterial infection discussed above.

**(b) Cow Dung for Floor Finishing**

Traditionally, application of cow dung-local soil paste is performed on walls, floors, kilns, and earthen stoves as a finishing coat or cleansing coat across households in India. The finishing coat is considered a benchmark of daily floor maintenance in rural households. The wall finishing elaborated in Figure 2.2 is a low-cost operation usually performed by local women. They also incorporate aesthetic design using red mud and charcoal. Cow dung is used for the finishing purpose due to the small size of its particulates suspended within the ordure matrix compared to Equus dung. In this region, a volume ratio of 1:2 of fresh cow dung and soil is uniformly mixed. Further, water equal to the volume of the cow dung is added to provide rheological consistency.

The paste is manually applied on the dry surface of the wall as per aesthetic requirements of the household. While performing wall finish, the paste is applied from bottom to top as illustrated in Figure 2.2. The patches and cracks on the walls are also repaired using this immingle. A tint of water is also applied to obtain smooth finish on the wall.



**Figure 2.2:** Local women of western Rajasthan region performing periodic finishing on mud structure

### 2.2.2 Manufacturing of Off-White Cruse in Western Rajasthan

The skills to cast clayey soils and to rotate the wheel respectively are the most important requirement to make a cruse. This can be traced back from the Mesolithic which gave importance to pottery, settlements, and domestication of flora and fauna towards the west of the Vindhya [Agarwal, 2007]. Mehrgarh Period VI (Third millennium BC) in this area showcased large circular pottery kilns, with use of pebbles for maintaining heat for a longer period [Agarwal, 2007]. The new thoughts of storing energy in stones or clay ceramics and use of circular kilns have reemerged and this may help retrodict these age-old facts [Nigay *et al.*, 2017; Waked, 1986]. The off-white cruse or pot is traditional drinking water temperature reducer used across western Rajasthan [Roux, 2015]. The cruse and other pottery products are very much adaptive to arid zones to keep water cool, vegetables, milk and milk-based products for long time use in comparison to open environment storage. These water pots are manufactured from local soil, small pebbles and organic material such as sawdust, donkey dung etc. [Roux, 2015].

The western Rajasthan salty clay cruse or pot is available in various sizes and shapes but all are basically globular shapes with a small neck [DMG, 1960; Kaurwar *et al.*, 2017]. The capacity of these pots varies from 5-liters to 30-liters. Although the manufacturing of cruse in different parts of India is similar but there is diversity of method of use of materials (raw material, wheel, and tool), method (posture during throwing beating, detaching pot from wheel, and pot decoration), and design (base and neck), method of sintering (open firing or firing in kiln) [Lemonnier, 2013]. This is due to using of local resources, their availability to the area, traditional knowledge transmissibility, and demand of local people [Lemonnier, 2013].

Selection of clay is an inherited traditional skill and may vary depending on geological aspect and feasibility to mould while working and variation on porosity while sintering [Sikdar and Chaudhary, 2015]. In this region of Rajasthan, these water pots are known to be cruse from "Pachpadra" (a village of Barmer district of Rajasthan) due to its location of manufacture. The main constituent of Pachpadra cruse was local salty clay and donkey dung. However, these pots are now being manufactured at different locations including Mokalsar, Sathin, Banad, Salawas, Rudakali, Bandera, Ramasani villages of Jodhpur district of Rajasthan state, but it utilizes sawdust in place of donkey dung and tempering is usually done using granite or kalmishora (Bajari in the local dialect). To manage the consistency and cohesiveness of clay different organic materials including straw, animal dung, sawdust is used along with clay. Some types of clay do not contribute to cruse manufacturing but used for decoration of cruse and for plastering the kiln [Sikdar and Chaudhary, 2015]. The uses of decorated pots (in rituals) are very much popular in Rajasthan and Gujarat. Originally the manufactured claywares and devices from clay were sold using barter system and usually made during all rituals of traditional people in western Rajasthan [Roux, 2015]. However, the demand for these pots gets majorly affected due to the introduction of metal and plastic utensils due to their durability, long lifespan and low cost [Nambuthiri *et al.*, 2015].

## 2.3 APPLICATION OF RELATED TECHNOLOGIES FOR CLAY CERAMIC WATER FILTER MANUFACTURE

Water quality is the primary concern of most of the developing countries [Gadgil, 1998]. Some of these countries receive support from World Health Organization (WHO) and UNICEF to lower down the mortality rates caused by waterborne diseases in their regions. For example, Thompson, 2015 claimed that 70% of diseases in Ghana were due to insufficient water treatment infrastructure to provide drinking water to the inhabitants of the country. These are basically due to bacterial pathogens in water [Plappally *et al.*, 2011]. Table 2.2 present the serious problem of contaminated water in India. Thirty out of 33 districts in Rajasthan are affected by fluoride contamination [Kalkoti, 2013]. According to BIS norms when the concentration of fluoride in water is above 1.5 mg/l it may lead to the health problem if consumed without treatment. In addition to this, high concentration of iron (above 1.0 mg/l) and nitrate (above 45 mg/l) in groundwater have been observed in Rajasthan. The people of rural Rajasthan walk on average a distance up to 5-8 kilometre to fetch water [Rinzin *et al.*, 2011]. Remedy for such drinking water woes can be a ceramic water filter [Plappally, 2011].

**Table 2.2:** Villages in the different states of India without sterile drinking water facility [Source: Kalkoti, 2013]

State	No. of districts	No. of villages	State	No. of districts	No. of villages
Andaman Nicobar	2	501	Maharashtra	35	41095
Andhra Pradesh	23	26613	Manipur	9	2199
Arunachal Pradesh	16	3863	Meghalaya	7	5782
Assam	8	25124	Mizoram	8	707
Bihar	22	39032	Nagaland	8	1278
Chandigarh	16	19744	Pondicherry	4	90
Chandigarh	14	23	Punjab	17	12278
Delhi	12	158	Rajasthan	32	40699
Haryana	19	6764	Sikkim	16	450
Himachal Pradesh	9	17495	Tamilnadu	30	15400
Jammu and Kashmir	12	6417	Tripura	4	0
Jharkhand	4	29354	Uttarakhand	71	15761
Karnataka	27	27481	Uttar Pradesh	38	97962
Kerala	14	1364	West Bengal	19	37955
Madhya Pradesh	48	52117			

These ceramic water filters are plagued with a multitude of issues too. They have very slow filtration rates [Plappally and Lienhard, 2013]. The maintenance and reliability of these filters depend on the user. The operation and maintenance of these filters are plagued with non-technical, cultural and social aspects of the society using these filters at a particular location. Brittleness of the clay ceramic becomes a major problem by increasing probability of fracture during transportation from one location to other. Hence manufacturing of these, at the point of use is to be sought. These filters due to their geometry and environmental characteristics require regular removal of bio-film and filtered impurities. Quality control during manufacture is observed as a big issue [Dies *et al.*, 2013]. Several other ceramic water filters are available in Indian market such as candle filters and ceramic disc. These are plagued with issues such as the biofilm formation and reliability issues due to a large number of connections. "Potters for Peace" advocates a factory mode of production for pot shaped ceramic water filters [Lantagne, 2001; Oyanedel-Craver and Smith, 2008]. A factory mode of ceramic water filter manufacturer may supposedly alter the traditional household pottery followed by the different societies in India. The manufacturing, as well as processing aspects of factory mode, may deprive and alienate traditional potters in India from there sustained social individual household business structures. Therefore, an indigenization of manufacture and processing is to be carried out to manufacture ceramic water filter within traditional potter households.

The filters may vary in shapes and size according to the manufacturing process, the material used, sintering method capacity, and flow rate. The compositional aspect plays a very important role to ensure ease of manufacturing, functional efficacy, strength characteristics, and less energy intensity. A brief detail of technologies used around the world has been presented in Table 2.3. The recent ethnographic study of material-based sustainability of pottery and study on compositional aspect of animal dung impregnation in soil for rural construction present significant scope of modification on ceramic water filter manufacturing in a very effective way [Kaurwar *et al.*, 2017; Roux, 2015; Satankar *et al.*, 2018]. Few points may be considered for indigenization of the existing filter manufacturing thought process and methods popular around the world. The compositional imbalance may result in poor bond strength between matrix and fibers on sintering [Lantagne and Clasen, 2012; Plappally, 2010].

**Table 2.3:** Popular ceramic water filter technology around the world

Geometry	Ingredients	Function	Other key variables
		Microbial removal (Potters for Peace pot filter, TERAFIL (Disc/Candle)	
(candle element) (India, Nepal)	Kaolinite+sawdust	Arsenic removal (Three Kalshi filter for arsenic (in Nepal), MIT Kanchan filter or Biosand Filter (Nepal and India)	grog, sawdust, sand
Pot (Potter for Peace)	Salty clay (India)+ Agriculture residue  Smectite+Flour/tea leaves/rise husk	Taste and odor removal  (Brita Filter)	wheel, hand mold, or mechanical / manual press

Non-uniform mixing of clay and organic material may also lead to early failure of ceramic water filter during use [Plappally, 2010]. To avoid this situation, step by step processing of clay and sawdust for pot manufacturing is to be carried out. Similar practices used for water jar manufacture can be used for water filter manufacture too. Addition of salt can be continued during manufacture of these filters. The water filter composites are initially dried before baking. Further, baking of filters may be accomplished using the household furnace. However, the functional difference between cruse and water filter may need change with baking method, temperature, time required and fuel consumption.

## 2.4 MANUFACTURING OF GREEN COMPOSITES

In general, the change in mechanical, structural, and functional properties of a specific material (matrix) can be obtained either by changing internal structure (bonds or atomic arrangement), by varying compositions, heat treatment processes or by introducing additives as reinforced materials [Soboyejo, 2002]. The present case emphasize study on green composites with age old use and practice in rural building constructions are wattle and daub, cob, rammed earth, adobe, compressed earth block (CEB) made using some reinforce material [Deboucha, 2011; Jaquine *et al.*, 2009; Maheri *et al.*, 2005; Shaffer, 1993; Smith *et al.*, 2000]. Reinforcements are termed as fibers, particulates, flakes, or whiskers [Soboyejo, 2002]. The source of these types of reinforced materials may be either natural or synthetic. The resulting property of the modified matrix is of immense importance. Particulate reinforced composites have particles with dimensions that are said to be almost equal in all directions. They may be of regular or irregular geometry [Campbell, 2010]. These composites are said to be weaker and less stiff than fiber-based composites, but considered cheap to common populace [Campbell, 2010]. Particulate composites contain reinforcement (0 to 50 by volume percent only) due to difficulties processing and their brittle nature [Campbell, 2010].

Rule of mixture theory (constant strain and constant stress rules) is widely use to estimate average mechanical properties (such as elasticity and strength) and physical property (density, thermal conductivity, diffusivity etc.) of composites along different compositions of matrix and reinforce material [Soboyejo, 2002]. This also signifies that this theory can also be useful to estimate the effect of locally available reinforcement materials when added to the soil-based composites. This theory had been used by Reaumur to assess the improvement in density and decrease of volume produced in a mixture of water and alcohol by mutual penetration [Christie, 1789]. The generalized expression of rule of mixture as presented in Eq.(2.1) can be used for wide range of loading conditions of stresses and strains, determining physical and mechanical properties of composite materials. This can be represented by Eq.(2.1), where  $X_{com}$  property of interest,  $m$  is a number between +1 and -1, subscripts mat, and fiber denote matrix and fiber respectively [Soboyejo, 2002].

$$(X_{com})^m = V_{mat} (X_{mat})^m + V_{fiber} (X_{fiber})^m \quad (2.1)$$

The green composite plays an important role in local waste management process. It can be considered as an economic and ecological advantage in addition to the significant improvement and value addition to the material properties [Thakur, 2013]. The green composite uses plant or organic waste (generally in dry condition) as reinforcements in soil. This offers homogeneous mixing of both the components [Kalia *et al.*, 2011]. The green composites referred in this thesis are particulate composites mixed in dry or semi-dry state.

## 2.5 MANUFACTURING METHODS OF TRADITIONAL CERAMICS

Manufacturing of the green composites precedes the ceramic manufacturing process. Water pots (off-white pots of Marwar), other pottery products are first moulded as a green composite before sintering them to the ceramic stage. The clayey soils and organic materials which are locally available are used by traditional potters as raw materials for manufacturing their products. Manufacturing of pots is a very seasonal process and is not carried out during rainy and summers periods in western Rajasthan [Vincentelli, 2000]. As far as the baking of pots and other pottery products is concerned it has traditionally been done using two firing techniques e.g. open firing and in firing using a kiln [Ravi *et al.*, 2007]. Out of these two techniques it open firing is oldest and still popular among potters of different parts of India [Ravi *et al.*, 2007]. The kiln preparation and loading require 2-3 hours while the firing time depends on the type of firing method used. The firing is usually done in the night and then the ripe products are laid in the furnace until next morning for cooling [Roux, 2015]. For an Asian potter, type of baking is a technical process choice rather than thought of design [Guo, 2017; Roux, 2015]. These procedural choices correspond to the local cultural traditions, climate, hereditary knowledge inertia, and social narratives related to local deities [Guo, 2017; Roux, 2015]. The technological changes are more resistant to adoption in domestic manufacturing than decorative styles [Habicht-Mauche, 2006]. Potter women (in India) inherit and transfer the materials knowledge on the soil, mixes additives, and the transport phenomena of clay [Kramer, 1997].

### 2.5.1 Open Firing

Bonfiring method is popular in several countries including Nigeria, North America, Palestine, Guatemala, Zaire, and Namibia [Gosselain, 1992]. Many potters of western Rajasthan region still use open firing technique to bake their products [Ravi *et al.*, 2007]. In western Rajasthan, they referred as "nyayo" in local *Marwari* dialect [Roux, 2015].

The shape may be circular as shown in Figure 2.3 with multiple hearth or triangle with single hearth. The firing process is usually done on a flat surface where the raw products are arranged in a circular pattern over a bed of dried chopped grass or similar materials from agriculture waste or animal waste. Agriculture waste such as twigs leaves are used as a fuel in this technique [Roux, 2015]. This firing provides temperatures of 500 °C to 850 °C [Vincentelli, 2000]. This technique did not leave much waste and hence less technological evidence of its practices [Vincentelli, 2000]. There are three distinct variants of open firing prevalent in India [Saraswati, 1978]. These are,

#### (a) With apical (apex) fire channel

Greenware is piled with their mouth inverted and arranged in circular form to take a dome shape [Saraswati, 1978]. While forming the dome shape the central portion is left vacant forming a vertical opening towards the top of the dome. The fuel is placed at the center and in between the layers of the greenware. The dome is finally plastered with a clayey soil and husk [Saraswati, 1978]. The top opening of



the dome is left un-plastered as hence used as a location to kindle the fire [Saraswati, 1978]. Duration for firing is about three to ten days, as per potter requirements.



**Figure 2.3:** Open firing (baking) of pots in Rajasthan [Source: Roux, 2015]

**(b) With horizontal (radial) fire channel**

Here the greenware is piled in radial rows at the base in a circular shape [Saraswati, 1978]. Narrow passages between the radial rows have an angular shape and act as locations of fuel deposition. These fuel depositions are fire channels which are diametrically opposite to each other and open towards its ends. These ends are stoked holes where fire may be kindled [Saraswati, 1978]. A number of stoke-holes will be double the number of radial rows of greenware piled [Saraswati, 1978]. Fuel may be arranged in between the radial lines and in between the greenware piles. The duration of the whole process is completed within half a day or a day.

**(c) Without any fire channel-**

This arrangement is shown in Figure 2.3. The greenware arranged again in circular form over a bed of cow dung and plant material but in a flat manner with no more than two layers. The piled greenware is covered with bamboo, soil paste, shards of cruse such that draughts of cool air is prevented from entering or conservation of energy occurs. The fire is kindled from all the directions about the circular pile [Saraswati, 1978].

### 2.5.2 Wall Firing

The technique of wall firing was promoted by Rajasthan government in 1995 but this technology could not become popular and now only a few potters use this technique. This method is a slight modification on open firing technique in which the brick wall is surrounded by the open firing place. The wall is opened on 3 to 4 side for firing [Roux, 2015].

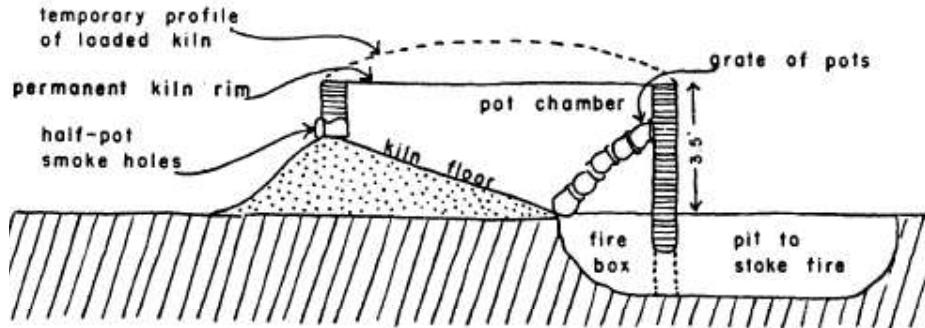
### 2.5.3 Pit Firing

Shallow pit method of open firing is oldest method of clay baking aging more than 1000 decades as shown Figure 2.4 [Ravi *et al.*, 2017]. This method of firing (with covering using grog or shards) is mostly used in India, while pit firing without shard (broken pieces of ceramic) is popular in Namibia and Sudan [Gosselain, 1992]. The furnace is made of the mud and cow dung mixture-based structure and a kind of deep trench of size 5 m deep and 3 m diameter [Gosselain, 1992]. Cow dung helps retain high temperature for a long time hence these structures are used widely in India [Arnold, 1999]. In here, the greenware is piled up inside the pit with or without enclosures such that they are close enough. The piling of the inverted greenware starts near to the stoke-hole and circumferentially continues in rows towards the rear [Saraswati, 1978]. Clay props are raised such that fire does not directly interact with the greenware. Cattle dung and plant materials are stuffed into the greenware [Saraswati, 1978].

For sintering or baking, the furnace is stacked to a point well above and after that fixed with a covering of straw and mud. After nightfall, the sintering starts and proceeds until early morning [Foster, 1956]. In the initial warming stages, fuel which produces enough smoke is utilized (kindling fire at the



stoke holes) while quick burning smokeless fuel is used towards the end of the baking process (fire at the stoke holes is quenched while a fire at the center takes over) [Arnold, 1999; Saraswati, 1978]. Smoking or pre-heating the clay green-ware before high-temperature baking limits thermal shock breakage [Perryman, 2008]. With geospatial location, technique, fuel wood sources and plant or combustible dung materials vary. In Northern India, especially animal dung is smoked while Tamil Nadu and Rajasthan potters expose raw greenware to midday sun to partially bake it [Perryman, 2008]. To learn much more about the possible pyrolysis of wood the interested readers are requested to read Ravi *et al.*,2007.



**Figure 2.4:** Pit firing used in West Bengal, India [Source: Foster, 1956]

The force of the fire is ventured up for the last two hours. A little opening is then made from mud and shards (pieces of broken cruse) covering and a baked cruse is taken out. The condition of the first cruse tells the potter whether to terminate baking or additional time is required [Foster, 1956]. Assessments of the advance of the terminating are likewise made by looking through the smoke, its color and potter's experience [Foster, 1956]. At this stage pots can be heated till the rosy orange color of the shards, regularly spotted dark due to uneven termination [Foster, 1956].

#### 2.5.4 Updraft Kiln Firing

In western Rajasthan modified pit firing or domestic hearth technique was introduced in 1987 which they learned from Gujarat state [Roux, 2015; Vincentelli, 2000]. The kiln does not provide much higher thermal advantage than a bonfire [Perryman, 2008]. This method requires almost one-fourth of the total time required in open firing that is the reason this method is quite popular.



(a)

(b)

**Figure 2.5:** (a) Updraft kiln at Sar village, Jodhpur, Rajasthan, India (b) Experimental scaled down updraft kiln at IIT Jodhpur (old campus), Rajasthan, India

Basically, this Gujarati kiln is vertical updraft with two chambers called firing and heating chamber. This kiln is made of connected bricks using clay and animal dung paste. The firing and heating chamber are separated by used truck leaf spring blades. The kiln used in this region has internal diameter 1.25 times the height. While the firing chamber and heating chamber is made at a volume ratio of 1.75. The kiln is tied using a wire to improve its structural integrity.

Mostly potters used the kiln that can be used to bake 50 pots at a time. However, relatively large Rajasthan kilns can bake 150 water jars at the same time. It has one opening at bottom side to supply the fuel e.g. garbage, grass, cactus, cornstalks, wood, pine and other scrap wood, rubber tire, dung chips and even diesel [Roux, 2015]. This method of firing is also similar to those used in other countries i.e. Mexico, Pakistan, Palestine, Egypt etc. [Hauge *et al.*, 1994]. During heating, the green-ware experiences structural change with a change in temperature as the fuel is added to the furnace. With raising temperature, the moisture from the green ware is driven out by evaporation. Crystal formation (quartz etc.) starts at approximate 350 °C. Until 350 °C the green ware undergoes many thermic variations both at the inside and outside surface. Further, at a temperature near 450 °C sawdust and other organic materials burn off and thus the pottery product becomes porous [Gosselain, 1992; Hauge *et al.*, 1994]. The coloration and intermittent black spots appear on the final produced cruse at 800 °C [Perryman, 2008].

**(a) Traditional Kiln Construction for Baking Pots and Other Pottery Products**

The traditional updraft kiln is mostly popular in many parts of India for baking pots and other pottery products [Ravi *et al.*, 2007]. The step by step construction of a traditional Gujarati klin is shown Figure 2.6 and Figure 2.7 and this was made by the Rajasthani potter (Mr. Farukh Khan, Sar village, Jodhpur) and the research team of IIT Jodhpur at Tindivanam in Tamilnadu. The spring leaves are used to provide a base to the pots while the portion below it kept for fuel burning. Once the kiln is constructed the periphery of it is surrounded by wires to avoid change in shape due to plasticity. The kiln is then allowed to dry in open environment till sufficient drying is achieved. The kiln is applied with a paste of animal dung and soil and is burned without pottery products before using it for baking.



**Figure 2.6:** Measurements and land preparation for pottery furnace construction





**Figure 2.7:** Steps of updraught kiln construction for baking pottery products

## 2.6 MICROSTRUCTURAL CHARACTERIZATION TECHNIQUES

### 2.6.1 Scanning Electron Microscopy (SEM)

The scanning electron microscope (SEM) is used for the examining and analysis of topology, morphology and elemental detection (with energy dispersive spectroscopy (EDS) attachment) of samples under observation [Zhou *et al.*, 2006]. This is a widely used technique of material characterization in scientific research and manufacturing, coal industries, forensic science, material science, image processing of clay etc. [Jones *et al.*, 1992; Liu *et al.*, 2011; Maire *et al.*, 2001; Postek, 2016]. In the present thesis SEM will be used to examine the elemental composition and surface morphology of the different horse dung-based composites and off-white pottery. SEM primarily consist of electron beam generating and accelerating system (electron gun), vacuum system (mechanical or diffusion pumps with vacuum controlling unit), a focusing and scanning system, a specimen stage, an electron detecting and multiplying system, amplifiers and CRT for visual inspection and imaging [Aharinejad and Lametschwandner, 1992]. Unlike microscopes, the source of specimen illumination is an electron beam here. The path length is small in case of an electron beam, hence whole set up is placed in a vacuum chamber at  $10^{-6}$  to  $10^{-10}$  torr [Suryanarayana, 2011]. Electromagnetic lenses focus the electron beam on to the sample material. The signals from the material surface are detected by an electron detector.

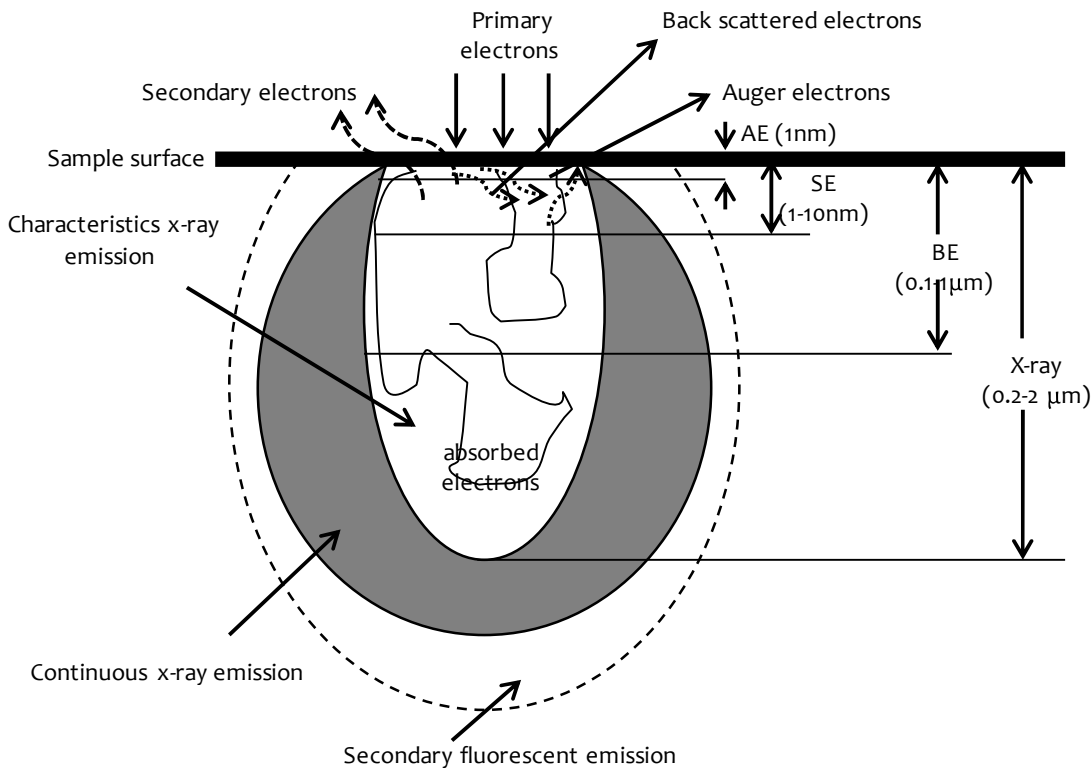
A negatively charged tungsten filament heated to about or beyond  $3000\text{ }^{\circ}\text{C}$  produces thermionic emission of electrons [Goldstein *et al.*, 2003]. The electron beam may have densities in the order  $5 \times 10^4 \text{ Am}^{-2}$  [Suryanarayana, 2011]. Still higher densities can be obtained when field emission sources (tips with 50-100 nm) are used. Strong electric fields at this tip extract electrons using tunnelling at low temperatures. Hence this source can be named cold cathode. The volume of material imaged and information generated from that volume is according to the energy of the produced electron [Goldstein *et al.*, 2003, Suryanarayana, 2011]. Therefore, produced electrons need to be accelerated through a potential difference. SEMs use around 1-50 V potential difference for providing electron an energy. The size of the image depends on the current that passes through the electromagnetic lens named condenser lenses. The high current passing through the lens provides smaller focal length and in turn produced large divergence. It is to be kept in mind that divergence of the electron beam is a function of the focal length of the lens. High divergence may

mean better control of beam current. The electron beam that was generated by the electron source to pass through the lenses to impinge the material surface is named primary electron beam.

The diverged or defocused electrons from condenser lenses may reach objective or probe lens. This lens focuses on the sample material. Information from the material may now be collected from the inelastic scattering of the electron beam from the material surface by the electron detector. Elastic scattering of electrons may also occur due to diffraction from the crystal planes of the material surface. The in-elastically scattered electron beams are used to produce the imagery which is used to assess chemical composition of the material specimen. The different signals or types of electrons that shown in Figure 2.8.

**(a) Imaging Using Secondary Electrons**

The interaction between the primary beam and loosely bound electrons in the atoms on the sample material surface help in the formation of the secondary electron [Goldstein *et al.*, 2003]. The angle between in primary electron beam and surface topography determined the secondary electron beam yield. The new electrons thus formed per primary electron is termed secondary electron emission coefficient and are used to provide imagery and information regarding the topography of the sample surface [Suryanarayana, 2011]. The energy of these newly formed electrons is not dependent on the energy of the primary electron beam and material under study. Any roughness or protruding surface increases secondary electron yield and hence detection. The low energy of this new electrons causes easier deflection of these electrons by the applied low potential difference and therefore may be collected with great effectiveness. The surface feature influences the number of electrons that reach the secondary electron detector from any point on the sample material surface under study. This electron intensity variation helps create the image with effective contrast with a surface that reveals the surface morphology.



**Figure 2.8:** Signals produced during SEM scanning of sample material surfaces [Source: Suryanarayana, 2011]

**(b) Imaging Using Backscattered Electrons**

Large angle or cumulative small angle scattering of the primary electron beam helps production of backscattering electrons [Goldstein *et al.*, 2003]. The proportion of electrons thus produced depends on the atomic number of the atoms specific to the sample material surface or phases with varied material compositions (atomic number contrast occurs) like alloys or ceramic, surface inclination, energy of primary electron beam or number of outer shell electrons of a specific atom or chemical element. For high atomic

number material surfaces, many electrons may get back-scattered with less waste of incident beam energy. Low atomic number surfaces may help generate a smaller number of electrons with large energy wastage. Ceramic or alloy phases with average high atomic number appear to be brighter while low atomic numbers may appear darker in the scanned imagery.

Information retrieval from x-rays by knocking out electrons from distinct shells (K, L, M etc.) within an atom of a specific element by a high energy incident electron beam causes generation of x-rays with wavelength depending on the character of the shell [Suryanarayana, 2011]. Therefore, chemical information can be retrieved by studying the characteristic of these rays emanating from the sample material surface. The different ways are in which the x-rays can be utilized are

#### (c) Elemental Maps Using X-Rays

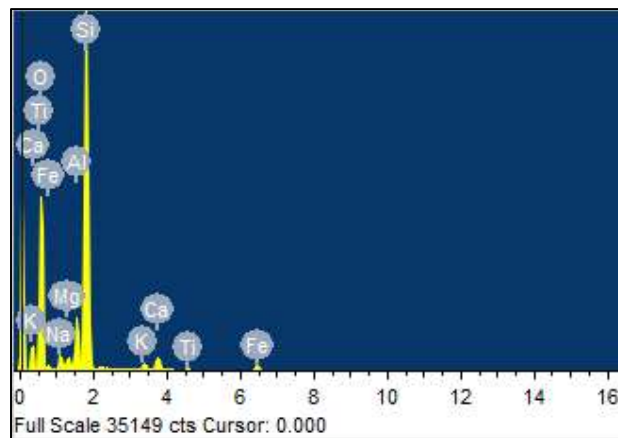
Here the characteristic x-ray photon detected is recorded as a color-coded dot (white, black or colored). Several elements may be detected on a sample surface [Goldstein *et al.*, 2003].

#### (d) Variable Pressure SEM

In this non-conducting sample study, the molecules of air together create a dense cloud of positive charge around the electron beam of the electron itself [Goldstein *et al.*, 2003]. This sample positivity neutralizes the negative electrons collected on its non-conducting surface. Imaging is performed on this specimen when the chamber pressure reaches a level such that most of the electrons are near to the specimen surface.

#### (e) Energy Dispersive Spectroscopy

An energy dispersive spectrum is illustrated in Figure 2.9. In this method, x-rays characteristic to a specific location on the sample material surface are generated, when the incident electron beam is stationary [Suryanarayana, 2011]. Figure 2.9 provides a graph of the intensity of the x-ray beam (number of x-ray photons detected) on the y-axis to the corresponding energies on the x-axis. Therefore, knowing the energies will help determine the chemical constituent responsible for helping emit such energy intensities. It is easier to detect high atomic number elements. The chemical analysis is obtained from a depth of 1-10  $\mu\text{m}$  from the sample material under study.



**Figure 2.9:** Sample EDS image of clay-sawdust mix used in cruse manufacture in western Rajasthan [EVO SEM, IIT Jodhpur]

#### (f) Field Emission-SEM (FE-SEM)

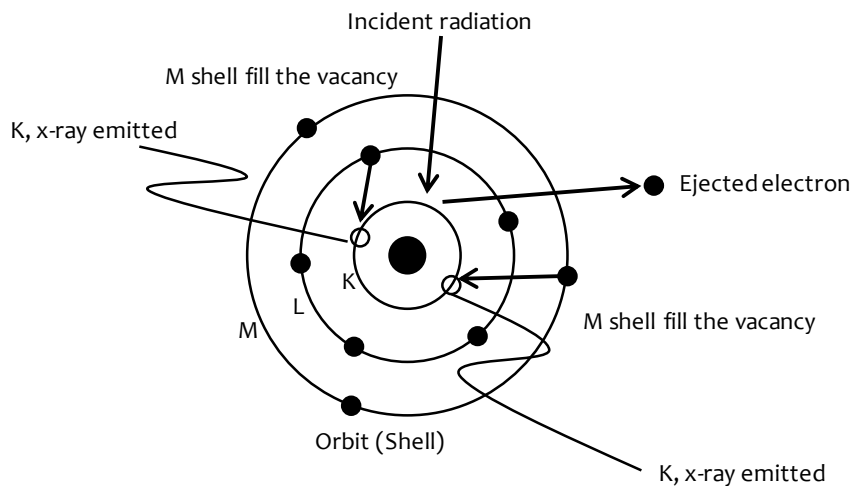
The filament setting is important while working with SEM for different applications to get optimal information. The working environment may cause electron scattering and contamination of microscopic column. Overheating of the filament may also occur. Thermal emission (i.e. tungsten filament) will occur to generating electrons. In practice, a magnification of about 100,000x is adequate for most samples [Goldstein *et al.*, 2003]. This cold field emission (FE) gun extracts electrons from the filament (cathode using an electrical field applied at the sharp tip). The electron extraction results in a higher electron yield and small beam sizes. This also helps provide a brighter signal along with a better resolution. The low accelerating voltages may be advantageously used for getting high-resolution imaging. Therefore, FESEM

is widely adopted for examining nano-materials, polymers, and thin film or coatings along with many non-conductive materials without a conductive coating. While preparing samples the specimen should be free of organic residues, oils, greases etc. They should also stable in a vacuum and should not change under the impact of the electron beam. Electro-static charge builds up over their surfaces is also prevented.

The problem due to charging gets aggravated in clay ceramics and non-conducting materials (Equus ordure composites) under study. One important solution for such problem is provided by operating SEMs at low voltages [Suryanarayana, 2011]. Other solution may be coating the non-conducting surface with a thin layer of the electrically conducting film [Suryanarayana, 2011]. Some SEMs do carry a conducting tape attached to the sample holder. Sometimes the samples are sputter coated with an electrically conducting film to optimize contrast within the image. Due to this some interference is also observed in the imagery, hence the studies performed towards this dissertation did not sputter coat the ceramic and composite samples. Porous samples were degassed before they were kept for scanning electron microscopy.

### 2.6.2 X-Ray Fluorescence Spectroscopy (XRF)

X-ray fluorescence are particularly well-suited for bulk chemical analysis of major elements. The ground fine powdered sample is used in most of the cases. As shown in Figure 2.10 an x-ray beam interacts with the electrons present in the inner shells of the atoms in a sample [Suryanarayana, 2011]. Since x-ray beam is of higher energy than the bond energy of the electron's displacement of the atoms from one shell to another shell occurs. The difference in energy between the primary x-ray beam emitted from the analyzer and the corresponding bond energy electrons in that orbits are recorded to get information on the sample composition. X-ray fluorescence (XRF) can widely be used in different areas of application including civil, mechanical, mining, glass manufacturing, geological, and environmental studies. The XRF is a time saving, easy and stable method over other methods of chemical analysis requires only low-cost sample preparation [Suryanarayana, 2011]. Electrons in distinct shells carry specific energies that decide their positions in an atomic shell. At the same time, the spacing between the orbital shells of an atom is also unique to the atoms of each element [Suryanarayana, 2011]. During interaction with a high energy beam, electrons may get knocked out and leave vacancies which makes the atom unstable. The atom must immediately correct the instability by filling these vacancies. The vacancies thus created can be filled from orbit at upper energy levels. An electron drops from an upper shell to an electron shell near to the nucleus loses energy. This energy difference determines the distance between them. The amount of energy lost in the process is unique to each element. Similarly, the individual fluorescent energies detected are specific to elements that are specific to the samples. In order to determine the quantity of each element present, the proportion in which the individual energies appear can be calculated by the instrument or by other software.



**Figure 2.10:** X-ray fluorescence by electron transfer due to high energy incident radiations

The previous section explains the working principle of scanning electron microscope (SEM) equipped with energy dispersive spectroscopy (SEM-EDS). In principle SEM-EDS uses an electron beam while XRF uses an x-ray beam. Both of these techniques can be used for qualitative (elemental detection)

or quantitative (concentration levels the entire element present in the material sample) analysis of a material including soils [Goldstein *et al.* 2003]. The results obtained are represented in ppm (parts per million) to percentages. SEM-EDS measure the elemental distribution on a material surface but sample preparation is complicated and time-consuming. In contrast, XRF analyzes the material sample without any specific pre-treatment [Shintaro *et al.*, 2013]. The case study discussed in this thesis uses both of these techniques for identification of elemental composition of samples distinct compositions of clay and organic materials.

### 2.6.3 Fourier Transform Infra-Red Spectroscopy (FTIR)

Fourier transform infrared spectroscopy (FTIR) is an analytical technique used to virtually analyze organic and inorganic materials samples including natural fibers [Fan *et al.*, 2012]. This can be used for a variety of applications including identification of unknown materials, qualitative analysis of the sample and chemical ingredient presents in the sample. The FTIR instrumentation unit contains an infrared source, interferometer beam, split detector, moving mirror, and a computer. In principle when a material sample is brought in the contact of IR radiation, the absorbed IR radiation (which represents sample characteristic at the molecular level) excites molecules into a higher energy state. Each microstructure of the internal material (atomic level) is capable to produce a unique infrared spectrum for specific material. Therefore, material identification can be done for almost all types of materials analyzing the peaks. The results are then presented as a plot of intensity (light transmittance or absorbance) versus wavelength after analysis [Salih, 2012] as shown in Figure 2.11.

The source emits the infrared energy which passes through a slit that controls the energy supplied to the sample. The beam enters the Michelson interferometer which works as an optical modulator that modulates the radiation wavelength (spectral encoding) that has been emitted by IR source [Salih, 2012]. The beam encounters with the sample and get transmitted or reflected off of the sample. In the end, the beam passes through a detector for final measurement that measures the reflected light. The intensity of the same is a function of its wavelength. The infrared spectrum is measured at high speed and the spectral range is continuously calibrated with a laser [Fan *et al.*, 2012; Salih, 2012]. The signal obtained from the detector is (interferogram) are digitized and analyzed with a computer for sophisticated data processing techniques [Fan *et al.*, 2012] where Fourier transformation takes place on the infrared spectrum.

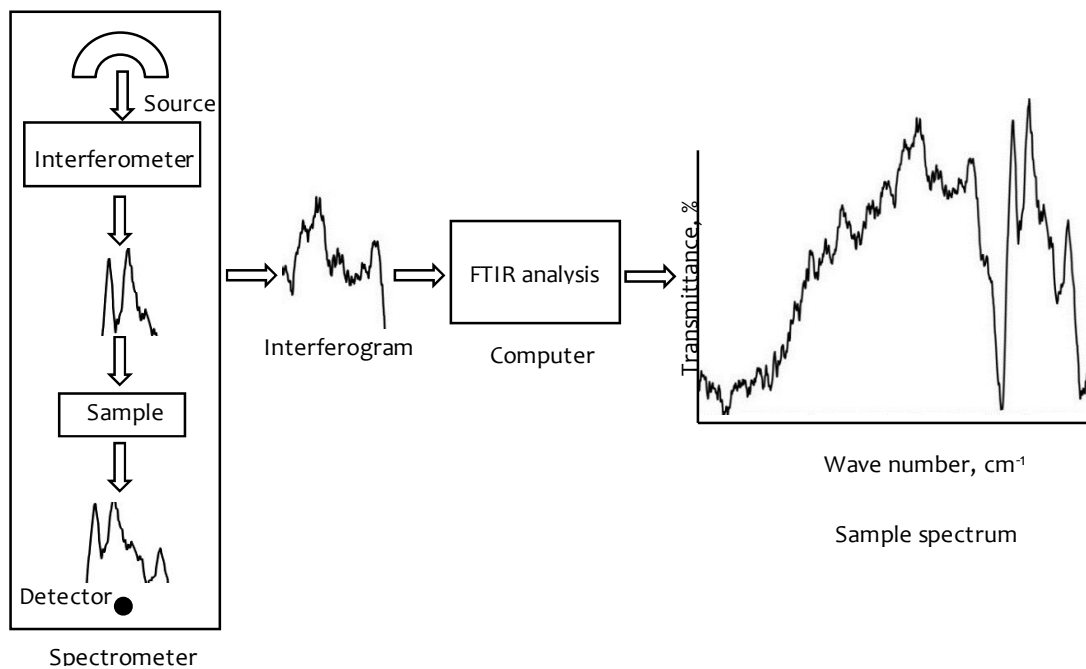


Figure 2.11: Outline of the Fourier transform infrared spectroscopy (FTIR) setup



## 2.7 MECHANICAL TESTING

The tests conducted within the thesis are towards assessment of physical properties of composites and ceramics from western Rajasthan (Chapter-3, Chapter-4 and Chapter-5). The basic raw materials used for their manufacture are local-pond-clayey soil with variable volumetric quantity of organic content (animal dung or sawdust) depending on their use. Measurement of strength in these composites and ceramics is important to ascertain reasons of its sustainability. These materials are influenced by different types of loads according to their location of function [Strong, 2008]. For example, a cruse used for (compressive) water storage activities but fails due to tensile stress application. The following types of standard tests are conducted to study the effects of the different loading.

### 2.7.1 Flexural Strength Test

bending test is an indirect means to measure flexural behavior of materials (clay and organic material-based composite in present case) [Mesbah *et al.*, 1999; Soboyejo, 2002]. The sample is generally rectangular or cylindrical in shape. The sample span is 75 cm or more [Plappally 2010]. On the basis of specimen loading the method is known as three-point bending (produces peak stress at mid-point and reduce at other places of the sample) [Soboyejo, 2002]. As shown in Figure 2.12 when the specimen is loaded monotonically, the specimen under test experiences tensile and compressive strength at the lower and upper surfaces respectively [Soboyejo, 2002]. The method of sample preparation, test procedure and calculation of flexural strength and fracture toughness is discussed separately in the subsequent chapters as per the requirements of the materials.

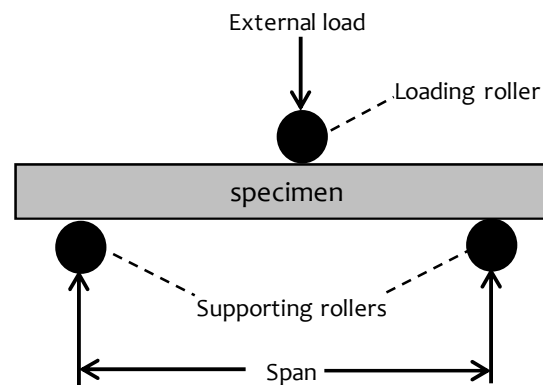


Figure 2.12: Three-point bend test bench

### 2.7.2 Compressive Strength Test

This test determines the behavior of various materials including metals, composites, ceramics, plastics, and concrete under axial compressive load. In test procedure the specimen (with small height to diameter ratio) is kept between the jaws and compressed. The deformation data is continuously recorded till the sample fails [Soboyejo, 2002]. The stress data recorded during the test is used to plot and draw the conclusion on strength of the material.

### 2.7.3 Fracture Toughness Test

Fracture analysis is performed using various means including measuring energy of failure, through stress analysis, mechanisms (micro or atomic) of fracture, and computational approaches etc. [Afendi, 2008; Mustapha *et al.*, 2015]. It is difficult to assess material characteristics from material failure criteria. The energy absorbed and plastic deformations are random in brittle materials. The evaluation of strength properties is performed using notched (pre-cracked) specimen. A three-point bending test as discussed in previous section can be used to fracture single edge notched beam (SENB) specimen to find the failure stress [Joudon *et al.*, 2014]. The stress intensity factor or fracture toughness predicts stress intensity near the tip of a crack caused by a distant load or residual stresses [Plappally, 2010]. There are three basic modes of crack-tip deformation, the opening (Mode-I), the in-plane shear (Mode-II) and the out-of-plane shear (Mode-III) as depicted in Figure 2.13 [Mustapha *et al.*, 2015; Soboyejo, 2002]. Stress intensity factor can be used as a measure of failure [Afendi, 2008].

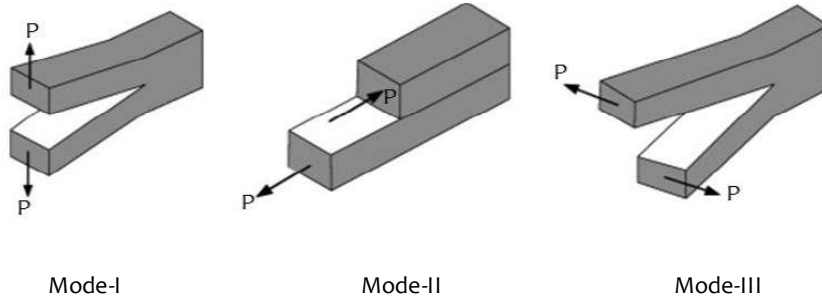


Figure 2.13: Modes of crack growth [Source: Feng et al., 2018]

## 2.8 STATISTICAL ANALYSIS

### 2.8.1 Statistical Variations Due to Manual Processing of Materials

All the methods of manual manufacturing by rural artisans (potters) always have high degree of randomness. Performance or functional effectiveness of a product is more important than quality for people in India. Traditionally, good quality water jars are identified by the acoustics generated when the sintered jar is tapped using hands softly on its external surface. The proper baking of a product is ensured by visual observation of the baked process. Similarly, the mud-based structures are advised to use cow dung paste finish to retain the functionality. This approach generally applied the use of a factor of safety [Haldar and Mahadevan, 2000]. The geometrical design configurations of the product are random and provide a probabilistic result for the integrity of the product. The approach of traditional practice for quality determination is only valid when same method of manufacturing is used.

### 2.8.2 Important Probabilistic Methods and Models

The normal distribution is an important distribution in order to model the properties of manually processed materials and structures. If the desired characteristics are randomly distributed, then a variable is the sum of the  $n$  number of independent variables [Salifu, 2015]. The properties of clay-based materials such as strength, density, curing, experience randomness because of manual processing in most of the cases and resulting property may get affected by various other parameters associated with it [Lumb, 1966]. The normal distribution may be used as tool for estimating design parameters in such cases [Lumb, 1966]. If material property  $x$  following a normal distribution can be represented as  $\sim N(\mu, \sigma^2)$  and the probability distribution of  $x$  can mathematically be represented as,

$$f(x) = \frac{1}{\sigma\sqrt{2\pi}} \exp\left[-\frac{1}{2}\left(\frac{x-\mu}{\sigma}\right)^2\right] \quad -\infty < x < \infty \quad (2.2)$$

Eq.(2.2) results in a symmetric unimodal or bell-shaped curve [Soboyejo, 2003] and  $\mu$  represents the mean value of while  $\sigma$  represents standard deviation. The probability density function  $f(x)$  may be integrated to obtain an expression for the probability of failure. Mathematically the cumulative normal distribution is defined by Eq.(2.3) as,

$$P\{x \leq a\} = F(a) = \int_{-\infty}^a f(x) dx \quad (2.3)$$

Eq.(2.2) may be evaluated by changing the variable and making mean and deviation independent by normalizing them by Eq.(2.4) as,

$$z = \frac{x-\mu}{\sigma} \quad (2.4)$$

$$P\{x \leq a\} = P\left\{z \leq \frac{a-\mu}{\sigma}\right\} \equiv \Phi\left(\frac{a-\mu}{\sigma}\right) \quad (2.5)$$

This process converts the distribution into standard normal distribution with cumulative distribution function presented in Eq.(2.5). Probability of survival can be calculated by subtracting the probability of failure from 1 [Soboyejo, 2003]. When the logarithm of desired characteristics of material

follows normal distribution then the material properties can be modeled using lognormal distribution function [Salifu, 2015].

The other distribution function used to predict the fatigue and strength character of brittle matrix composite is Weibull distribution function. It has also been extensively used to the model of time to failure of ball bearings, structural elements in aircraft and automobile components [Montgomery and Runger, 2010]. The probability distribution function of this distribution can be given Eq.(2.6) by,

$$f(x) = \frac{\beta_0}{\eta} \left(\frac{x}{\eta}\right)^{\beta_0-1} \exp\left[-\left(\frac{x}{\eta}\right)^{\beta_0}\right] \quad x \geq 0 \quad (2.6)$$

Where  $\eta (> 0)$  and  $\beta_0 (> 0)$  are called scale parameter and shape parameter respectively. The mean and variance of the Weibull distribution can be written by Eq.(2.7) and Eq.(2.8) respectively as,

$$\mu = \alpha_1 \Gamma\left(1 + \frac{1}{\beta_0}\right) \quad (2.7)$$

$$\sigma^2 = \alpha_1^2 \left[ \Gamma\left(1 + \frac{2}{\beta_0}\right) - \left\{ \Gamma\left(1 + \frac{1}{\beta_0}\right) \right\}^2 \right] \quad (2.8)$$

By selecting appropriate scale and shape factor cumulative distribution equation can be written in the form of Eq.(2.9)

$$F(x) = 1 - \exp\left[-\left(\frac{x}{\alpha}\right)^\beta\right] \quad (2.9)$$

Both of the probability distribution functions discussed above require a large number of experiments. When a large number of experiments (more than 30) is conducted. Then the tests are said to adhere to central limit theorem [Ang and Tang, 1975]. Normality in any data of required properties can be predicted by adhering to central limit theorem.

### 2.8.3 Multiple Regression Modeling

In the discussion above, the randomness of ceramics and composite properties were clear. Further, any response variable  $Y$  is observed to be dependent on several other random variables  $X_i$  due to manual mode of household manufacturing practiced by potters in India. It is also clear that property development  $Y$  depended mostly on the history of the process undergone by an immingle. This would mean a stochastic development of the ceramic or composite material is envisaged.

In such cases when the property of individual interest (defined in terms of mean and variance of the depended variable) depends on multiple independent variables, multiple regression linear modeling method is used [Montgomery and Runger, 2010]. Mathematically when dependent variable  $Y$  is normally distributed and it is function linear function of  $n$  variables  $X_1, X_2, X_3 \dots \dots X_n$ , the mean  $E(Y)$  and standard deviation  $V(Y)$  can be written by Eq.(2.10) and Eq.(2.11) respectively as,

$$E(Y) = \alpha_0 + \alpha_1 X_1 + \alpha_2 X_2 + \alpha_3 X_3 + \dots \dots \dots + \alpha_n X_n \quad (2.10)$$

$$V(Y) = \sigma^2 \quad (2.11)$$

The model constant  $\alpha_0$  regression coefficients  $\alpha_1, \alpha_2, \alpha_3 \dots \dots \alpha_n$  are required to be estimated using multiple regression analysis from given data set. To obtain the same point estimators  $\delta_0, \delta_1, \delta_2, \delta_3 \dots \dots \delta_n$  are calculated such that sum of squared residuals can be minimized. Mathematically this can be achieved using least square method approach. The estimated coefficient signifies the effect of individual parameter contribution in the development of dependent variable. The work covered in this thesis uses Minitab16 for mathematical modeling of sample data.

### 2.8.4 Theoretical Development of Empirical Models

In order to understand the physics of material characteristics of the newly developed composite material, a new stochastic multi-parameter approach has been developed [Ang and Tang, 2007; Haldar and Mahadevan, 2000; Haan, 1977]. Statistical formulation aims to identify groups of similar independent influence and to understand the influence boundaries of variable groups with their separations [Haan,

1977; Soboyejo 1965; Soboyejo, 1968]. Hence the development of composite and ceramic property  $Y$  assumed to be and expressed by Eq.(2.12) [Soboyejo, 1968; Soboyejo, 1973; Soboyejo *et al.*, 2001; Yadav *et al.*, 2015].

$$Y = F(X_1, X_2, X_3) \quad (2.12)$$

The predictor random variables  $X_i$  for  $i = 1, 2, 3$  of the response, random variable  $Y$  represent not only the major materials and mechanics variables which can affect this problem but also time. The stochastic multi-parameter mechanics model developed in the empirical studies which this thesis follows can be expressed by Eq.(2.13) as follows.

$$Y = Y_0 \prod_{i=1}^k X_i^{b_i} \quad (2.13)$$

**Table 2.4:** Example of statistical correlation between assumed predictor variables  $X_1, X_2$  and  $X_3$

Predictor Variables	$X_1$	$X_2$	$X_3$
$X_1$	3.7	0	0
$X_2$	0	3.7	0.933
$X_3$	0	0.933	2

Where,  $Y$  represents the response variable. For example, the weight loss at any particular instant of time. Assume that  $X_1, X_2, X_3$  are the first, second and third variable which are correlated as shown in Table 2.4.

#### (a) Multivariate Analysis

From Table 2.4 it was found that the predictor variables  $X_2$  and  $X_3$  were correlated and therefore not independent random variables. In order to remove the statistical correlations, the multivariate statistical analysis was applied to this problem. Here all the participating correlated variables defined with  $X_1, X_2$  and  $X_3$  can be regarded as a matrix  $[X_{ij}]$ , where  $i=2,2,3,\dots,k$  observations. This may be enumerated by Eq.(2.14) as,

$$[X_{ij}] = [\bar{X}_{i,2} \quad \bar{X}_{i,2} \quad \bar{X}_{i,3}] \quad (2.14)$$

where  $i=2,2,3,\dots,k$  and  $j=2,2,3$ . It should be noted that some information contained in one variable defined by a column vector  $\bar{X}_{ij}$  is also contained in other participating variable column vectors. The elements in the matrix  $[X_{ij}]$  are correlated and have a mean  $\mu_{X_{i,j}}$  and standard deviation  $\sigma_{X_{i,j}}$ . To get an unbiased prediction, the correlated predictor matrix column elements are linearly transformed and reduced as represented by Eq.(2.15) [Yadav *et al.*, 2015],

$$\frac{([\bar{X}_{i,j} - \mu_{X_{i,j}}])}{\sigma_{X_{i,j}}} = [T] \left[ \frac{[\bar{V}_{i,j}]}{\sqrt{\lambda_{i,j}}} \right] \quad (2.15)$$

This means that multivariate analysis starts from the correlated variables  $\bar{X}_{i,j}$  and uses eigenvalue  $\lambda_{i,j}$  and eigenvector  $[\theta]$  formulation of the original data matrix to derive new mathematically independent variables [Yadav *et al.*, 2015]. Here  $[T]$  is an orthogonal transformation square matrix of size  $n \times n$ , with eigenvectors  $[\theta]$  of (the covariance matrix) the reduced participating predictor variables. These new variables  $[\bar{V}_{i,j}]$  are the principal components (PC) or scores which are linear combinations of the original correlated variables  $\bar{X}_{i,j}$  weighted using the new independent column matrix [Wei *et al.*, 2012]. These PC elements express the correlation magnitude and positivity or negativity in which the original correlated variables contribute to the scores [Papachristodoulou *et al.*, 2006]. The covariance matrix  $[C^r]$  with reduced participating variables is written in the form of Eq.(2.16) [Haan, 1977].

$$[C^r] = \begin{bmatrix} 2 & \cdots & \rho_{\bar{x}_{2,j}} \\ \vdots & \ddots & \vdots \\ \rho_{\bar{x}_{i,2}} & \cdots & 2 \end{bmatrix} \quad (2.16)$$

Eigenvectors help in finding the variances or error in values of each participating variables. This is done by converting the covariance matrix of the participating predictor to a characteristic value problem. The analysis in Eq.(2.16) aims to compress the original space occupied by the dependent variable into a new independent variable space of reduced dimensionality, while retaining the independent properties and influences of the original variables [Ang and Tang, 2007]. The characteristic value problem is solved as shown by Eq.(2.17) as,

$$\left[ [C^r] - \lambda_{i,j} \begin{bmatrix} 2 & 0 & 0 \\ 0 & 2 & 0 \\ 0 & 0 & 2 \end{bmatrix} \right] [\theta_{i,j}]^T = 0 \quad (2.17)$$

It should be noted that the above matrix containing the normal standard deviations is a diagonal matrix and the diagonal elements are the characteristic roots of the equation and represent all the  $\lambda_{i,j}$ . The trace of the diagonal matrix denotes the sum of the variances of all the components in the transformation matrix and is termed the total system variance. The eigenvector matrix may be represented in Eq.(2.18) below as,

$$[\theta_{i,j}] = \begin{bmatrix} \theta_{2,2} & \cdots & \theta_{2,k} \\ \vdots & \ddots & \vdots \\ \theta_{k,2} & \cdots & \theta_{k,k} \end{bmatrix} \quad (2.18)$$

**Table 2.5:** Statistical correlation between dimensionless orthogonal new predictor variables:  $\bar{v}_1$ ,  $\bar{v}_2$  and  $\bar{v}_3$

Predictor Variables	$\bar{v}_1$	$\bar{v}_2$	$\bar{v}_3$
$\bar{v}_1$	1	0	0
$\bar{v}_2$	0	1	0
$\bar{v}_3$	0	0	1

This matrix is then normalized by dividing in each case the square of the eigenvector element  $\theta_{i,j}$  by the sum of the squares of the eigenvector elements  $\theta_{i,j}$  in  $i=1,2,3,\dots,k$  modes and then finding the square roots of the resulting quotients. This normalized matrix is the transformation matrix  $[\bar{T}]$  [Soboyejo, 1968; Soboyejo, 1965]. By substituting this transformation matrix  $[\bar{T}]$ , in Eq.(2.15), we get the orthogonal uncorrelated column matrix  $\bar{v}_{i,j}$  for  $j=1, 2, 3$  [Yadav *et al.*, 2015]. The above procedure produced the new multi-parameter multivariate solution to this problem. The Table 2.5 shows the new independent variables.

### (b) Lognormal Multi-Parameter Model Development

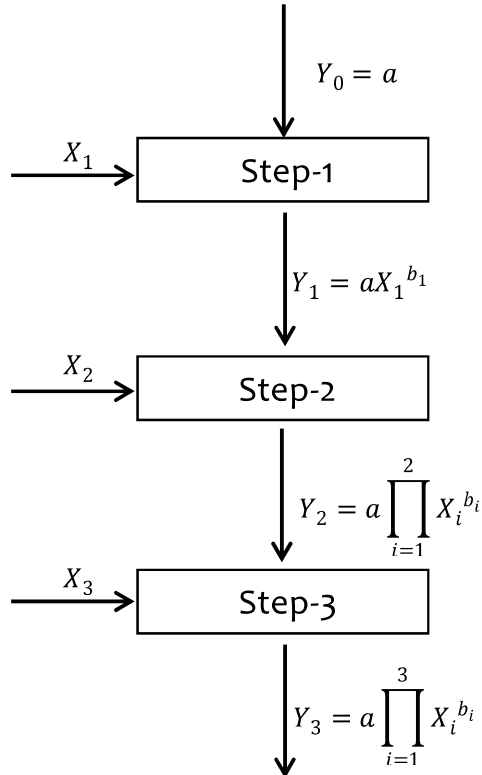
The basic nonlinear data of the response variable  $Y_i$  with a stochastic nature can be expressed in the form of Eq.(2.19) [Yadav *et al.*, 2015].

$$\frac{Y_i}{Y_{i-1}} = X_i^{b_i} \quad (2.19)$$

The above expression of  $X_i^{b_i}$  for  $i = 1,2,3..$  are the transfer functions of the stochastic process model. This simply means that in order to advance from step  $i - 1$  to step  $i$  or for the stochastic model to grow from step  $i - 1$  to step  $i$ , the model needs to have a transfer function  $X_i^{b_i}$  where,  $X_i$  is predictor random variable at step  $i$  and  $b_i$  is a model constant applicable at step  $i$  [Haan, 1977]. This explains the stochastic or zero memory nature of the stochastic process model [Plappally, 2010].

The transfer function from step 0 to step 1 is,

$$\frac{Y_1}{Y_0} = X_1^{b_1} \quad (2.20)$$



**Figure 2.14:** A stochastic lognormal model with variables  $X_i^{b_i}$

The transfer function from step 1 to step 2 is,

$$\frac{Y_2}{Y_1} = X_2^{b_2} \quad (2.21)$$

The transfer function from step 2 to step 3 is,

$$\frac{Y_3}{Y_2} = X_3^{b_3} \quad (2.22)$$

At step 1,  $i=1$ , we have

$$Y_1 = Y_0 Y_1^{b_1} = a X_1^{b_1} \quad (2.23)$$

Where  $a = Y_0 =$  initial value of the stochastic process model, and which is a model constant in this problem.

At step 2,  $i=2$ , we have,

$$Y_2 = Y_1 Y_1^{b_2} = a X_1^{b_1} X_2^{b_2} \quad (2.24)$$

At step 3,  $i=3$ , we have,

$$Y_3 = Y_2 Y_2^{b_3} = a X_1^{b_1} X_2^{b_2} X_3^{b_3} = a \prod_{i=1}^3 X_i^{b_i} \quad (2.25)$$

$$y_i = \ln y_i = \ln a + \sum_{i=1}^3 b_i \ln x_i \quad (2.26)$$

It can also be expressed as

$$y_i = \bar{a} + \sum_{i=1}^3 b_i x_i \quad (2.27)$$

The above steps (from Eq.(2.20) to Eq.(2.27)) show the stochastic formulation of this problem; where  $a, b_1, b_2$  and  $b_3$  are model constants. To enhance the predictability of formulation in Figure 2.14, the response variables can be modified to a quotient response variable [Plappally, 2009]. Quotient response can be assuming the largest mapping of a specific variable  $X$  from the set of influencing variables  $X_i$  for  $i = 1, 2, 3 \dots n$  to reduce the response variable to a very small value [Soboyejo, 1973]. The reduced variable  $\frac{X_m}{Y_i}$  becomes the new response variable  $q_i$  [Plappally, 2010]. Mathematically it can be written in the form of Eq.(2.28) as,

$$q_i = F(X_1, X_2, X_3 \dots X_n) \text{ where, } q_i = \frac{X_m}{Y_i} \text{ for } i = 1, 2, 3 \dots n \quad (2.28)$$

### 2.8.5 Kolmogorov-Smirnov Test for Distribution

It is one of the popular goodness of fit test also called as KS-test. In this test experimental cumulative frequency and theoretical distribution function are compared and decision on rejection or acceptance of theoretical model is taken. For a given sample size if large discrepancy is observed, the model is considered rejected.

In this test maximum difference between the empirical distribution function  $S_n(x)$  and hypothesized distribution function  $F(x)$  over the entire range is the measure of discrepancy between the theoretical model and the observed data. Let this max difference be denoted by  $D_n$  and Tang, 1975 by Eq.(2.29) as,

$$D_n = \max|F(x) - S_n(x)| \quad (2.29)$$

Theoretically  $D_n$  is a random variable whose distribution depends upon the number of variables  $n$ . For a defined level of significance say  $\alpha$  this test compares this maximum difference [Ang and Tang, 1975] by Eq.(2.30).

$$P(D_n \leq D_n^\alpha) = 1 - \alpha \quad (2.30)$$

The critical values at the various level of significance and fixed and defined for different values of sample size. If observed  $D_n$  is less than the critical value. The proposed distribution is accepted or otherwise would be rejected.

## 2.9 THE SOFTWARE TOOLS AND PACKAGES

### 2.9.1 Minitab

Minitab is a statistical software. Descriptive statistics including mean, standard deviation, quartiles can easily be calculated for large data set. Multivariate framework including factor analysis, principal components and eigenvalues can be plotted using loading diagrams and score plots. Normality of empirical data can be assessed using histograms. Correlation and covariance analysis between participating variables can be easily accomplished using Minitab 16 (Minitab 16, IIT Jodhpur Licensed)

### 2.9.2 Origin

The Origin 8 is used in this document to showcase x-ray diffraction plots. This software package and analysis tools include different features that can be used to perform peak analysis (as shown in Figure 2.15) of data for material characterization, statistical analysis, 3D surface mapping and image processing. These characteristics make Origin 8 suitable for graphical representation and analysis of data in different fields of material engineering.



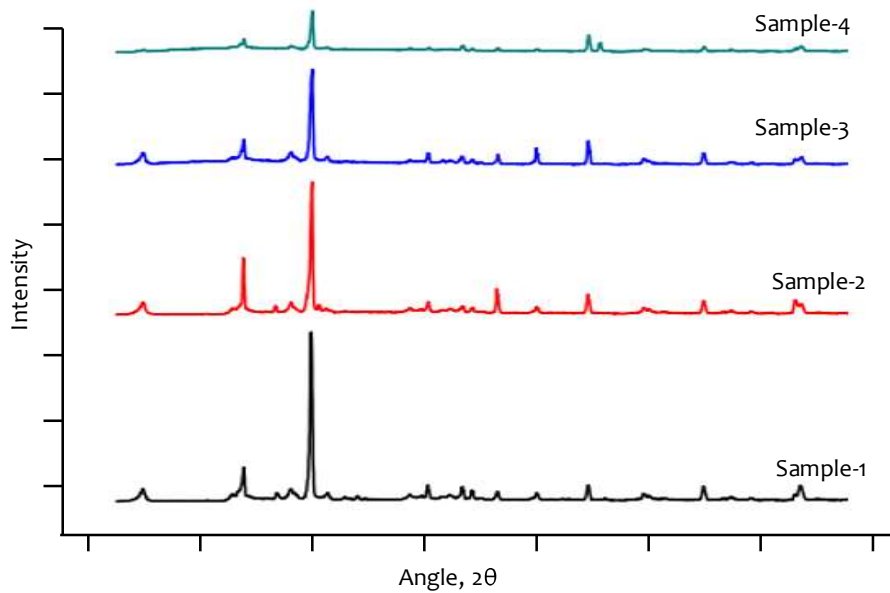


Figure 2.15: Sample stacked plot XRD data using Origin software package

### 2.9.3 CES Edu Pack

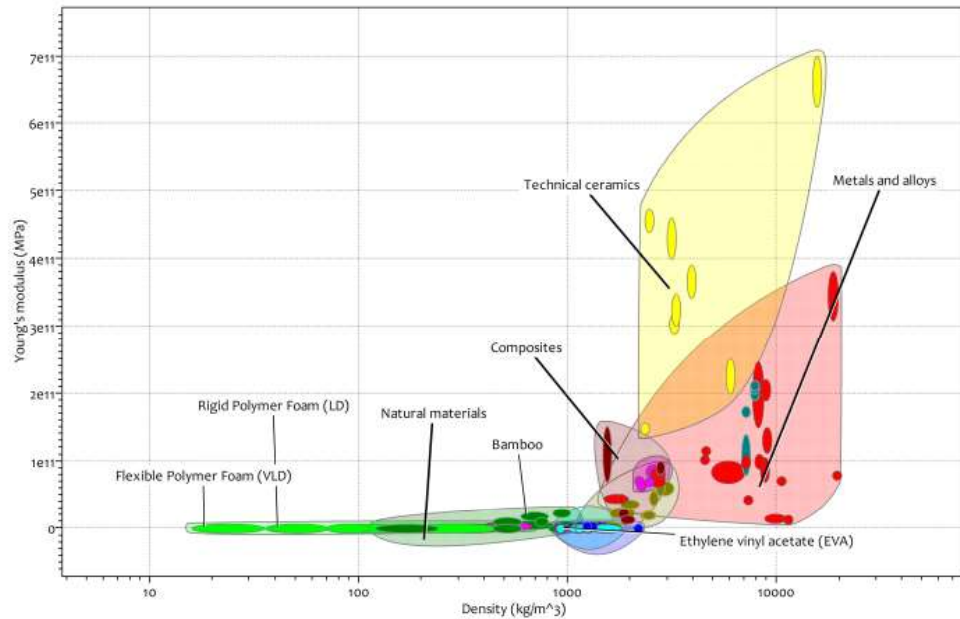


Figure 2.16: Sample plot between Young's modulus and density by CES Edupack software

CES EduPack software package help to improve the understanding of materials by virtual representation the material properties in various combinations of designing, manufacturing, economic and environmental point of view. This provides the flexibility to choose the material for a particular application. Basically, it comprises of a complete database of materials (all commercial materials of all category). The information is generated in graphical format (as shown in Figure 2.16) after comparing across the parent group of material or processes possible with that material. Material selection and matching the properties of new materials to the existing materials to compare changes may also be carried out using this software package.

### 2.10 SURVEYS

In this thesis study of technological skill-sets inherent to specific communities is performed. Therefore, a face to face interaction with the members of the community practicing these skills is considered

pertinent. Study of socio-technical systems stemming from the epistemological point of view is an emerging field. The field may impact health, production, transport, education, markets and mobility [Erez *et al.*, 2014]. For example, from the literature survey it is clear that ecological niche of the *Marwari horses* may have favored the use of horse dung within specific households [Talhelm *et al.*, 2014]. Similar findings where technology get embedded with a very co-operative notion as a socio-technical system will link immediate environment with local society and should define boundaries for certain location specific innovations [Henrich, 2014; Henrich, 2015]. Here socio-behavioral structured queries were developed and constructed for administration in villages in western Rajasthan.

The survey was in English or Hindi and translated into *Marwari* (oral) before being administered to potters at Banad, Jhalamand, Sar, and Salawas village in Jodhpur. Health professionals and *Anganwadi* workers reviewed the question for meaning, alignment towards the drinking water health and cultural understanding. The interview questions were first piloted and posed before people from *Prajapati* community (Hindu potter community personnel at Arna Jharna Desert Museum, Jodhpur) before administration. Gender and age of the potter family members and their level of influence in manufacture were requested during the demographic part of the survey. Level of education was also ascertained. The survey form is attached in the Appendix.

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