



Full length article

Effect of high temperature on some physical properties of bentonite from Barmer, Rajasthan, India

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ABSTRACT

Globally bentonite clay has been proposed as an engineered barrier material for safe underground disposal of high-level nuclear waste. Clay has many favorable properties such as high liquid limit, and plastic limit along with other properties which make it the most suitable for this application. In the present study, an attempt has been made to study the behavior of Barmer bentonite under the influence of high temperatures up to 120°C. Properties of Barmer bentonite namely, liquid limit, plastic limit, and maximum dry density have been determined after thermal treatment at 25°C, 60°C, 80°C, 100°C and 120°C. The extensive experimental results indicate that liquid limit and plastic limit show a decreasing trend while maximum dry density increases with an increase in temperature. Liquid limit and plastic limit decrease up to 12 % and 11 % respectively when the temperature reaches up to 120°C. Maximum dry density increases by 10 % due to thermal treatment and optimum water content decreases by up to 4 %. Statistical analysis has been carried out to obtain the correlation between temperature and physical properties of Barmer bentonite such as liquid limit, plastic limit, maximum dry density, and optimum water content. The XRD analysis of Barmer bentonite at room temperature and 120°C shows very small variation in mineralogical composition. Whereas, interlayer distance has been measured and found to be decreasing with an increase in temperature. Further, a comparative analysis shows that the measured properties of studied Barmer bentonite lie in the range of previously measured values of other types of bentonite across the globe.

1. Introduction

Bentonite has been widely proposed as a suitable engineered barrier material in a deep geological repository for the safe disposal of high-level nuclear waste. The concept of deep underground disposal of hazardous nuclear waste is based on multiple engineered barrier systems such as natural engineered barrier systems and engineered barrier systems. In the deep geological repository, host rock and surrounding material are termed as natural engineered barrier whereas backfill and buffer for metallic canister can be grouped under engineered barrier system. Bentonite under a highly compacted state constitutes favorable thermo-hydro-mechanical-chemical properties for buffer material (Jefferson and Rogers, 1998; JNC, 2000; Villar and Lloret, 2004; Abuel-Naga et al., 2006; Onal and Sarikaya, 2007; Grim and Guven, 2011; Yoon et al., 2024; Jha et al., 2025; Jaiswal et al., 2025). As per the proposed design for an underground waste repository, a sealed metallic

container with nuclear waste will be placed in an array of designed boreholes. Further, bentonite clay is to be used to fill the gap between the sealed waste container and pit wall. Nuclear waste disposal program of different countries consists of granite as a suitable host rock and bentonite as buffer material. It has been hypothesized that the buffer will be fully saturated after a certain time of its post-closure phase. Hence, engineered barrier material must minimize the flow of water into the repository and radionuclides from the repository under increased temperature conditions. Extensive experimental analysis has been carried out by several researchers (Hueckel and Baldi, 1990; Lloret and Villar, 2007). on thermo-hydro-mechanical behavior of clay as engineered barrier material in different countries. The study of physico-mechanical behavior of bentonite at higher temperatures attracted several researchers across the globe.

Zhang et al. (2004) studied the effect of drying (air and oven) on liquid limit, plastic limit, and plasticity index of soil and found that

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drying at low temperatures also reduced tested parameters. The major reason behind such a change could be the diffuse double layer (DDL). DDL is a critical factor which controls the plasticity of soils because it causes a change in the amount of moisture attracted and absorbed to the surface of clay minerals. The soil plasticity is highly influenced by the particle's external surface, pore water characteristics, and cation exchange capacity (CEC).

In the recent past, studies on Atterberg limits and their relationship with the external surface of soil particles have been also carried (Hammel et al., 1983; Khorshidi et al., 2017) and found that there is a strong dependency on Atterberg limits on the external surface. The surface area and DDL thickness of soil largely depend on the presence of a type of minerals, salt concentration in pore fluid, and CEC. The thickness of DDL varies proportionally with change in temperature whereas the surface potential reduces for a specific amount of surface charge. On the other hand, it can also be explained as the DDL thickness varies with the thickness dielectric constant, i.e. thickness of DDL increases when the dielectric constant increases. The surface potential of soil particles increases with a decrease in dielectric constant particularly when the surface charge is constant. Hence, it can be observed that an increase in the temperature of soil will cause a reduction in the thickness of the diffused double layer and an increase in the surface potential of soil/clay particles (Mitchell, 1993; Abu-Zreig et al., 2001).

The mechanism of drying of soil can be categorized in, chemical, physico-chemical, and mechanical. The change/loss of the diffuse double layer causes the variation in adsorption and absorption layers and it occurs due to physico-chemical phenomena. The chemical process causes the precipitation of soluble salts present in pore water. Pore water with a high concentration of soluble salts results in the cementation of particles during the precipitation of soluble salts at particle contact points. Sometimes these newly created contacts become insoluble. The process of cementation during precipitation is irreversible and this will lead to the growth of particle size and decrease the soil plasticity (Zhang et al., 2004; Rao and Ravi, 2013; Estabragh et al., 2016).

Temperature in the near field area in the underground repository for the disposal of nuclear waste may rise to 100°C in the repository (Cho et al., 1999; JNC, 2000). Such a rise in temperature may alter its physical behavior.

The temperature in the near-field area of an underground repository for nuclear waste disposal may rise up to 100 °C. Such an increase in temperature can alter the physical behavior of the bentonite. The present study focuses on investigating the effect of elevated temperatures (up to 120 °C) on Barmer bentonite from Rajasthan. The treatment duration was carefully selected to obtain the thermal effects, with 24 h

considered sufficient to ensure complete thermal exposure of every bentonite particle, allowing property changes as per the applied temperature.

2. Material and methods used in the study

Bentonite from Barmer, Rajasthan-India has been used in the study and acquired commercially. Barmer bentonite has been supplied as a pulverized powder (Fig. 1a). Bentonite is a type of earth material in which smectite is the most dominating mineral. Smectite can be termed as a group of minerals which predominantly constitute sodium and calcium montmorillonite. The molecular structure of bentonite is composed of 2-layers of silica tetrahedral and one layer of alumina octahedral (Zeng et al., 2022).

The current study was exclusively focused on determining the effect of temperature on the physical properties of Barmer bentonite. In the present study, different physical properties of bentonite such as liquid limit, plastic limit, plasticity index, maximum dry density, and specific gravity have been determined by extensive experimental work.

In the present study, bentonite has been kept at different temperatures for more than 24 h (Fig. 1b) so that heat can penetrate up to every grain of bentonite soil. Experiments have been carried out to determine the different properties after the soil reaches room temperature. Each test has been done more than three times to ensure the accuracy of the result for each property. For saturation of Barmer bentonite distilled water has been used in the study.

3. Results and discussion

3.1. Liquid limit and plastic limit at different temperatures

The liquid and plastic limit (Atterberg limit) of soil represents the transition between different states such as liquid state, plastic state, and brittle solid state of soil (Casagrande, 1958). Terzaghi noticed that these parameters potentially consist of important information regarding the behavior of soil. Casagrande (1958) developed a standard testing method for liquid and plastic limits which was modified using the falling cone penetration method. In this study, falling cone penetration method (IS 2720- part 5, 1985) has been used to determine the liquid limit of bentonite at different temperatures. Soil sample for the test has been prepared as per standard, 150 gm. of bentonite with a suitable amount of distilled water has been mixed and kept for 24 h in a sealed container (Fig. 2b) to ensure proper mixing of soil and distilled water. After 24 h the sample was filled in a 50 mm diameter cup and the cone was allowed to free fall (Fig. 2a). As per standard, the cone should



Fig. 1. (a) Barmer bentonite clay used in the study and (b) thermostatically controlled oven for heat treatment.



Fig. 2. (a) Liquid Limit testing apparatus and (b) sealed container for sample preparation.

penetrate 20–25 mm in a soil-filled cup and three samples for each soil water mixture have been tested. Further, for moisture content, samples after each test have been kept in the oven at a constant temperature of 110°C. Plastic limit tests have been done after the liquid limit test by keeping the soil sample for some time to reduce the moisture content. For fast removal of moisture, wet soil is kept on a glass plate (Fig. 3a) and air is circulated using a fan. Further, the ball of wet soil has been rolled between the fingers with optimum pressure. Threads of wet soil up to a diameter of 3 mm were rolled until the fracture was observed (Fig. 3b). After this condition, the rolled thread was kept in an oven at a constant temperature (105°C) and plastic limit was obtained.

Another parameter, the plasticity index has also been determined at different temperatures. The plasticity index is the difference of the liquid limit and plastic limit (Fig. 5).

The effect of temperature on the index properties of Barmer bentonite has been analyzed. The liquid limit shows sensitivity towards the increase in temperature.

The liquid limit (Fig. 4), plastic limit, and plasticity index (Fig. 6) of Barmer bentonite have shown a decreasing trend with an increase in temperature. The change in the behavior against increased temperature is the result of the coalescence of soil particles to form larger particles. It has been observed by several researchers such as Blight (1988) and Fookes (1997), that the soil is sensitive to the drying phenomena even at low temperatures. The changes in physical properties and structure have been observed during the drying of soils even at ambient temperature (White, 2005). This change in the structure of soils results in variations of specific surface area of soil. Change in specific surface area

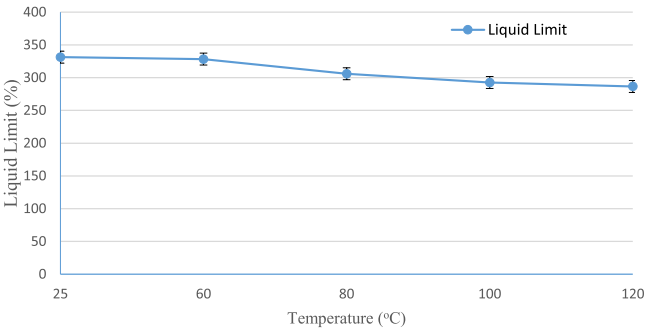


Fig. 4. Liquid limit of Barmer bentonite at different temperature.

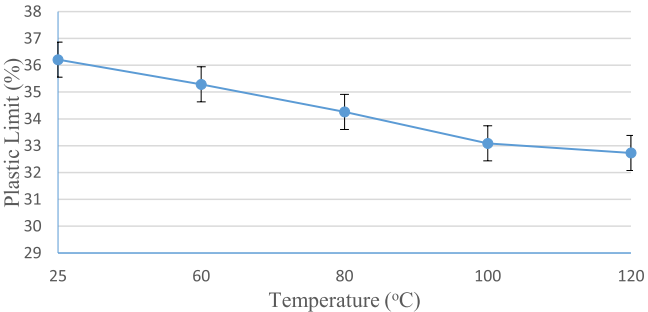


Fig. 5. Plastic limit of Barmer bentonite at different temperature.



Fig. 3. (a) Smooth glass plate for plastic limit test and (b) rolled samples.

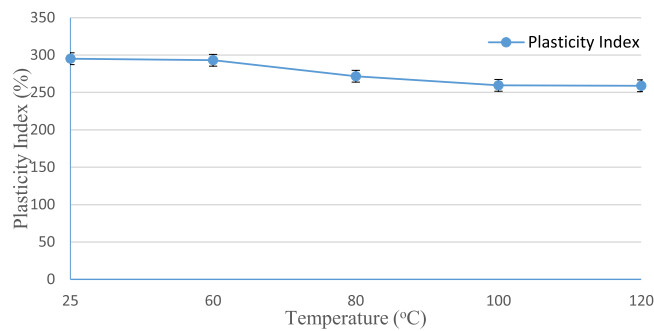


Fig. 6. Plasticity index of Barmer bentonite at different temperatures.

Table 1

Experimental result for Liquid limit of Barmer bentonite at different temperature.

Sample No.	Temperature (°C)	Liquid Limit (%)	Average Liquid limit (%)	Standard Deviation
BBT-1	RT	327.83	331.34	3.38
BBT-2		335.92		
BBT-3		330.33		
BBT-4	60	332.65	328.37	4.01
BBT-5		329.44		
BBT-6		323.01		
BBT-7	80	307.77	305.97	1.30
BBT-8		304.74		
BBT-9		305.42		
BBT-10	100	288.61	292.62	3.21
BBT-11		296.45		
BBT-12		292.81		
BBT-13	120	288.69	286.54	3.44
BBT-14		296.47		
BBT-15		289.81		

Table 2

Experimental results for plastic limit test of Barmer bentonite at different temperature.

Sample No.	Temperature (°C)	Plastic limit (%)	Average plastic limit (%)	Standard Deviation
BBT-1	RT	41.52	36.21	3.96
BBT-2		35.08		
BBT-3		32.02		
BBT-4	60	32.89	35.29	2.95
BBT-5		33.54		
BBT-6		39.45		
BBT-7	80	37.27	34.26	3.07
BBT-8		35.47		
BBT-9		30.04		
BBT-10	100	36.78	33.09	3.60
BBT-11		34.28		
BBT-12		28.21		
BBT-13	120	35.57	32.73	3.11
BBT-14		34.21		
BBT-15		28.40		

reduces the water absorbing capacity and hence, the liquid and plastic limit of soil. (Tables 1–3).

Atterberg limits of Barmer bentonite show considerable sensitivity towards temperature. The experimental results obtained can be divided into two zones; up to 60 °C and above temperature range. In the first zone, the change in liquid limit is comparatively small (< 1 %). As the temperature increases, the interlayer distance of Barmer bentonite is found to be decreasing using XRD analysis. It could be one of the prime reasons for such changes.

In the second region, above 60°C temperature, the change in specific surface area may occur which affects the water absorbing capacity of

Table 3

Maximum dry density at different temperatures.

Sample No.	Temperature (°C)	Maximum dry density (kN/m ³)	Average maximum dry density (kN/m ³)	Standard Deviation
BBT-1	Room	11.91	11.78	3.73
BBT-2	Temperature	12.16		
BBT-3		11.27		
BBT-4	60	11.79	11.82	0.97
BBT-5		11.96		
BBT-6		11.73		
BBT-7	80	12.51	12.76	2.58
BBT-8		13.12		
BBT-9		12.66		
BBT-10	100	13.28	13.16	0.87
BBT-11		13.09		
BBT-12		13.10		
BBT-13	120	13.04	13.18	2.93
BBT-14		13.58		
BBT-15		12.91		

bentonite and hence the liquid limit decreases. Another reason could be the formation of bigger size particles by joining two small-sized particles and cementation due to the precipitation of oxidized minerals. It affects the water absorbing capacity by preventing the penetration of water into the soil particles. Both phenomena cumulatively affected the liquid limit and decreased up to 291.66 %. The plastic limit of bentonite has been tested at different temperatures and it shows a decreasing trend with an increase in temperature. The plastic limit of bentonite decreases sharply up to 100°C from 36.21 % to 33.01 %. Beyond this temperature, the change in plastic limit is very small and reaches up to 32.73 %. The plasticity index also shows a similar trend to that of liquid and plastic limits.

3.2. Maximum dry density at different temperatures

The maximum dry density of Barmer bentonite has been determined as per Indian Standard (IS, 2720 Part 8, 1983). Bentonite clay (6 kg) has been mixed with initially 1.5 kg (25 % by weight) of distilled water and kept for 24 h. The soil-water mixture has been compacted in a 1000 cc volume cylinder in 5 layers to fill the cylinder (Fig. 7). The dry density



Fig. 7. Maximum dry-density test apparatus.

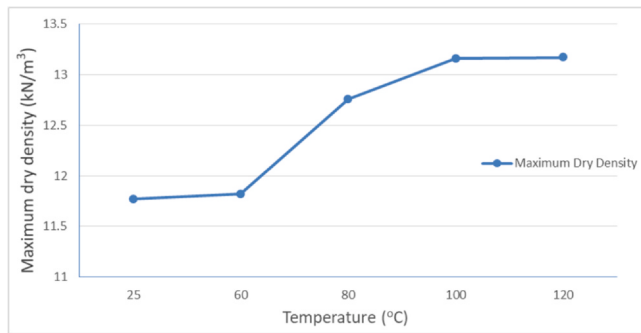


Fig. 8. Maximum dry density of Barmer bentonite at different temperatures.

of soil increases initially with an increase in water content but after a certain percentage of water addition, density starts decreasing and this is the point of maximum dry density at a given moisture content. For the determination of dry density, the weight of compacted soil with a base plate was observed. Three samples were tested for each temperature and a wet bentonite sample was taken after each of the three tests and kept in the furnace for moisture content determination. Further, water was added to the soil and kept for 24 h and a similar procedure has been carried out.

The maximum dry density of Barmer bentonite has been determined at different temperatures (Fig. 8). The values of maximum dry density have been found to increase with temperature. The results obtained can be grouped into three zones; zone-I, up to 60°C, zone-II, from 60°C to 100°C and zone-III, up to 120°C. For the first reason, maximum dry density increases from 11.78 to 11.83 kN/m³. Further heating of bentonite clay causes the increase of maximum dry density from 11.83 to 13.16 kN/m³. There is a small increase in dry density of bentonite has been observed above 100°C.

3.3. Optimum water content at different temperatures

The optimum water content has been determined at different temperatures (Fig. 9). Optimum water content represents the water content at maximum dry density of soil. Experimental results obtained can be divided into three zones; zone-1, room temperature to 80°C, zone-2 from 80°C to 100°C and zone-3 from 100°C to 120°C. In the first zone, the optimum water decreases slowly from 36.30% to 36.15%. Further heating of bentonite clay reduces the optimum water content rapidly and decreases up to 35.20%. Above this temperature, the optimum water content decreases slowly and reaches up to 34.84% at 120°C.

Specific gravity tests have been carried out as per Indian Standard (IS-2720-Part-3, 1980). A bottle of 50 ml capacity (Fig. 10) has been used to determine the specific gravity of Barmer bentonite 5 gm of oven-dried bentonite has been placed in bottles and kept for 8–12 h after adding distilled water to fully saturate the bentonite. Further,

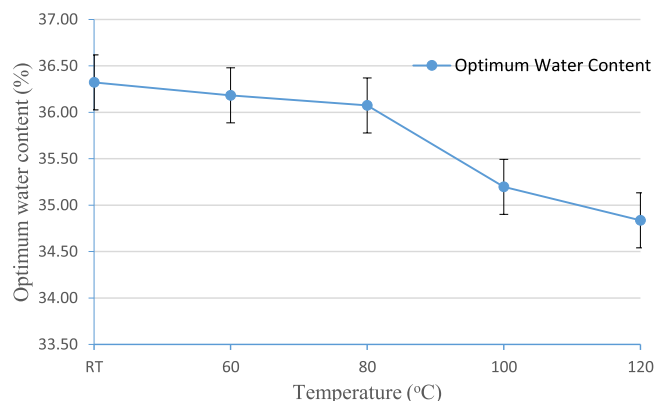


Fig. 9. Optimum water content of Barmer bentonite at different temperatures.



Fig. 10. Specific Gravity test bottles of Barmer bentonite.

distilled water was added to the neck of the bottle and mixed properly. Water baths have been given at a constant temperature of 27°C for 2 h to ensure that the temperature throughout the sample reaches up to 27°C. Each bottle has been weighed before and after every step of the experiment. The value obtained for specific gravity is 2.30.

The formation of the diffuse double layer (DDL) in soil takes place primarily on the external surfaces of soil and clay particles. The amount of moisture attracted and the thickness of the DDL depend largely on the surface area of particle. Studies on Atterberg limits and their relationship with the external surface of soil particles (Hammel et al., 1983; Zhang et al., 2004) have shown a strong dependency of Atterberg limits on the available surface area. The surface area and DDL thickness of soil are significantly influenced by the mineralogical composition, salt concentration in the pore fluid, and cation exchange capacity (CEC). The thickness of the DDL varies proportionally with temperature changes, whereas the surface potential decreases for a given surface charge. Alternatively, this behavior can also be described in terms of the dielectric constant, where an increase in dielectric constant results in a thicker DDL. Conversely, the surface potential of soil particles increases with a decrease in dielectric constant, provided the surface charge remains constant. Thus, it can be inferred that an increase in soil temperature leads to a reduction in DDL thickness and an increase in the surface potential of soil/clay particles (Mitchell, 1993; Khorshidi et al., 2017).

Physico-chemical phenomena due to heating may result in the cementation of clay mineral particles because of precipitation of Fe and Al oxides. Such cementation increases the particle size reduces the water-absorbing capacity of clay particles and increases the maximum dry density of clay. The effect of cementation is considerably low at lower temperatures and hence change is small at higher temperatures (zone II and zone III). Another factor, interlayer distance may also affect the maximum dry density of bentonite and hence, cause the reduction in water absorption capacity and result in an increase in maximum dry density. The decrease in water absorption capacity can also be related to the change in the Atterberg limit of Barmer bentonite at elevated temperatures.

XRD analysis has been done to observe the changes in distance between the layers of Barmer bentonite (Fig. 14). Detailed analysis of XRD results for interlayer distances indicates that with the increase in temperature the interlayer distance changes from 7.12 Å to 6 Å, when temperature increase from room temperature to 120°C. Such a change in distance between the layers could be the major factor for the changes in Atterberg limits.

4. Statistical analysis

4.1. Relation of liquid limit, plastic limit, and maximum dry density with temperature

Regression analysis has been done to establish the quantitative correlations between the properties of Barmer bentonite and temperature. The objective of statistical analysis was to obtain a mathematical

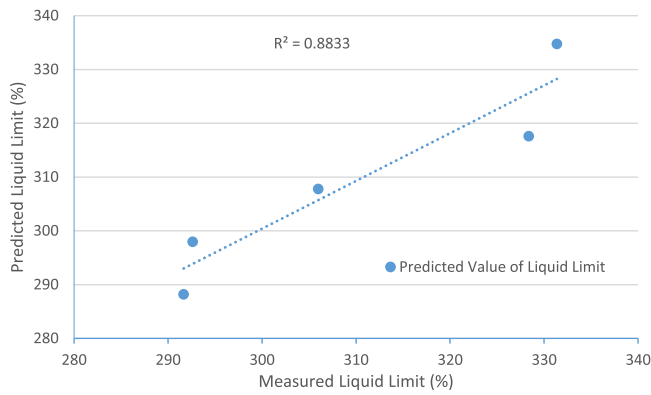


Fig. 11. Predicted liquid limit Vs. experimental liquid limit of Barmer bentonite.

functions/equation that may represent the variation of liquid limit (*LL*), plastic limit (*PL*), and maximum dry density (*MDD*) with change in temperature. The regression equations for *LL* and *PL* (Eqs. (1) and (2)) shows, negative coefficients for temperature, highlighting that both liquid limit and plastic limit decrease with an increase in temperature. In contrast, maximum dry density exhibited, positive correlation, validating that higher temperatures improves dry density. To validate the regression equation's predictive strength, experimentally measured values of *LL* and *PL* are compared with the predicted, by the regression equations (Fig. 11 and Fig. 12). The regression coefficient (R^2) has been used as a statistical indicator for the accuracy of regression equations. For *LL*, the R^2 value obtained as 0.9833, which shows an excellent fit between the statistical model and the experimental results. Similarly, for *PL*, the R^2 value also obtained as 0.9833, indicates a good predictive model.

$$LL = 347.54 - 0.48 * T \quad (1)$$

$$PL = 37.34 - 0.04 * T \quad (2)$$

The best-fit equation for the maximum dry density of Barmer bentonite has been also proposed (Eq. (3)). The R^2 value obtained is 0.8514 which indicates the goodness of the equation. The value (R^2) is comparatively lesser than that of the liquid limit and plastic limit. The coefficients of temperature have positive values which suggests that the increase in temperature results in the increase of maximum dry density. The values of maximum dry density have been predicted using the proposed equation and shown in Fig. 13.

$$MDD = 1.118 + 0.002 * T \quad (3)$$

The closeness of predicted trends and observed values shows the robustness of the regression equation/models. The high R^2 values indicates that the equations can be used for predicting the behavior of

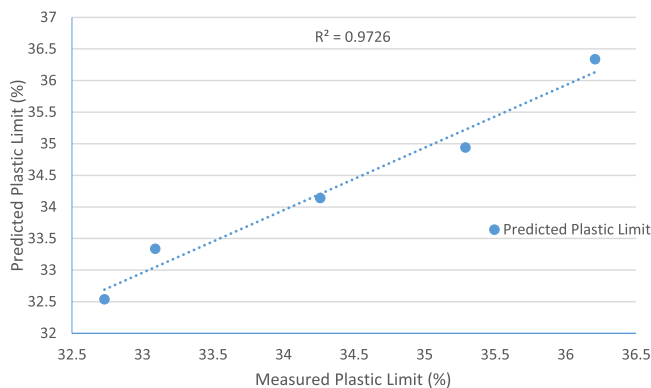


Fig. 12. Predicted plastic limit Vs. experimental plastic limit of Barmer bentonite.

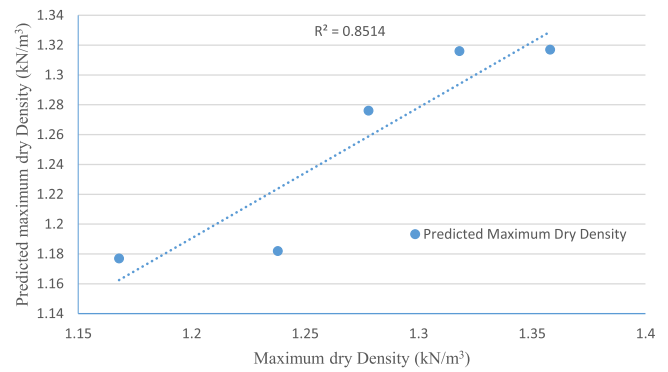


Fig. 13. Predicted maximum dry density Vs. experimental maximum dry density of Barmer bentonite.

Barmer bentonite under thermal loading. Such equations are important while designing a dedicated engineered barriers for nuclear waste repositories and other high-temperature applications.

5. XRD analysis

The change in temperature may cause the variation in the mineralogical constituents of the Barmer bentonite. In order to assess this variation the XRD analysis has been carried out. Bentonite samples at designated temperature has been taken carefully in a sealed glass container to ensure purity. Further, these powdered samples used for testing. XRD analysis of carefully collected and prepared bentonite sample has been done using a high-resolution X-Ray Diffraction (HR-XRD) instrument, Rigaku Smart Lab 9 kW, powder diffractometer equipped with a θ - 2θ goniometer. The typical instrument employs a Cu $K\alpha$ radiation source ($\lambda = 1.5406 \text{ \AA}$) having a parallel beam geometry and a graphite monochromator to ensure high spectral purity. XRD test conducted with a step size of 0.02° and scan speed optimized for resolution. Such configuration of equipment enables precise determination of microstructural evolution of bentonite under elevated temperatures.

The results obtained show that the measured constituent of the clay is montmorillonite (Fig. 14). It has been observed that there is a very small variation in the mineralogical composition of montmorillonite. Additionally, the measurement of interlayer distance has been studied by the XRD. Using Bragg's law (Pope, 1997) (Eq. (4)), the basal spacing observed is $7.12 \text{ \AA}$ at room temperature and $6.0 \text{ \AA}$ at 120°C . Changes in the interlayer spacing indicate the loss of interlayer hydration with the increase in temperature of Barmer bentonite.

$$n\lambda = 2d \sin \theta \quad (4)$$

Where d = d-spacing or interlayer or basal spacing, θ = Bragg's angle in radian, n = Parameter related to reflection and λ = X-ray wavelength.

6. Comparative analysis

Several types of bentonite have been analyzed for its application as a suitable material for engineered barrier systems. The properties obtained from different experiments conducted on Barmer bentonite have been compared with other bentonite across the world and India as well (Fig. 15). The properties of bentonite chosen for comparative analysis are GMZ bentonite (Zhijian, 2006), Barmer 1 (Rao and Ravi, 2013), FEBEX (Villar and Lloret, 2008), Kunigel VI (Komine, 2004), Avonseal (JNC, 2000) and Kyungju (Cho et al., 1999). The different properties have been compared at room temperature. There have been scares of thermo-physical data of bentonite at elevated temperatures. The comparison of properties indicates that the thermophysical properties of studied Barmer bentonite are in accordance with previous studies.

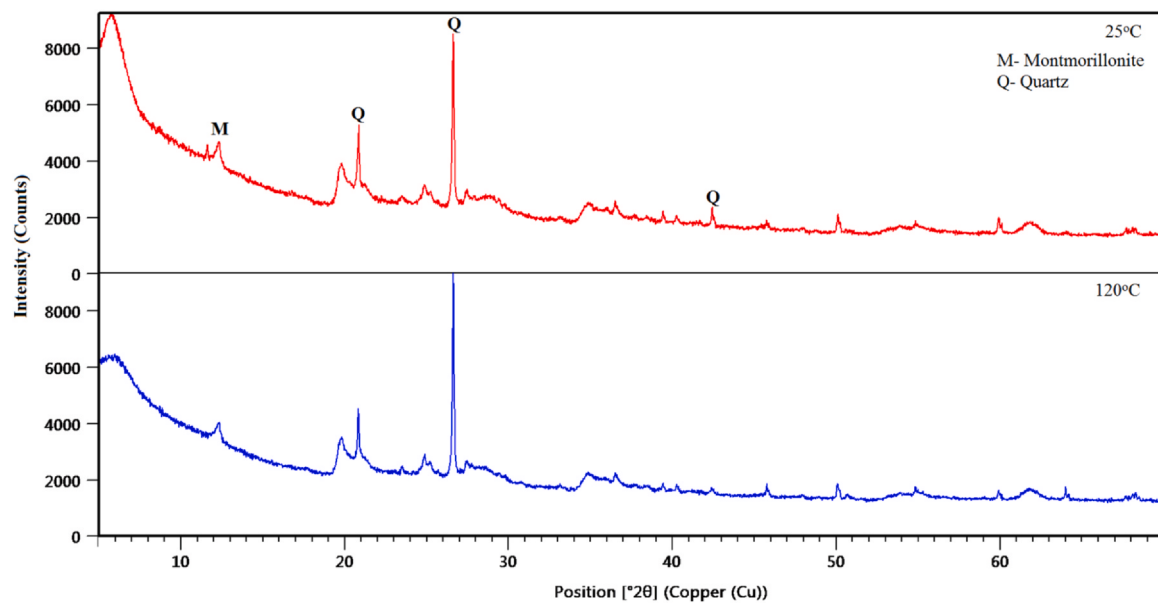


Fig. 14. Result of XRD analysis of Barmer bentonite at 25 °C and at 120 °C.

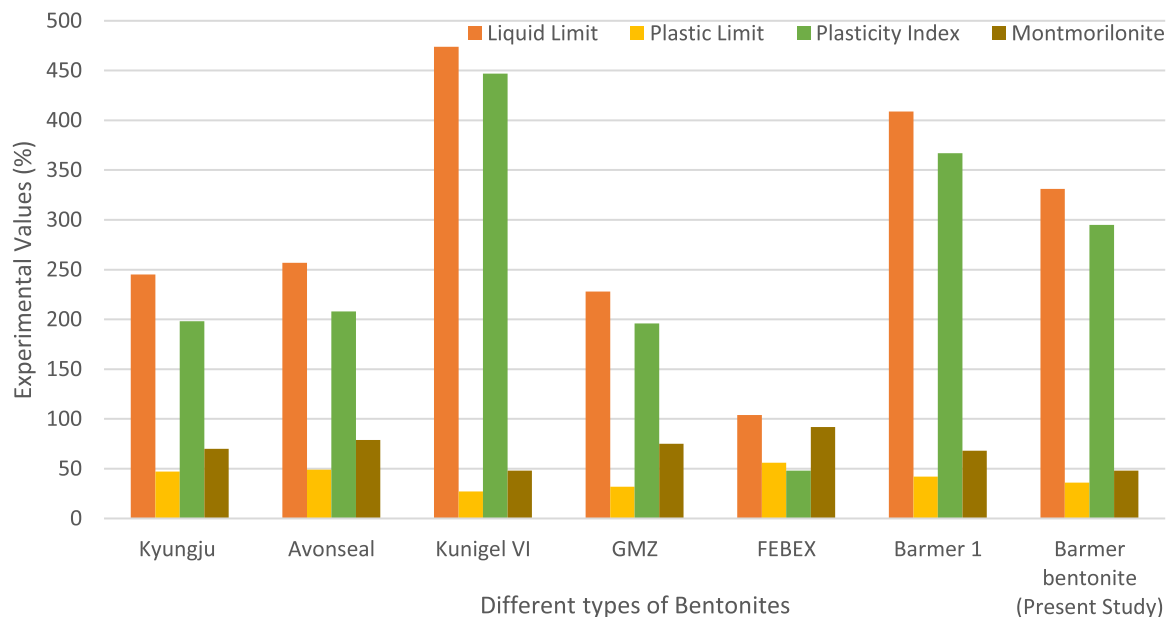


Fig. 15. Comparison of different properties of Barmer bentonite with other studies at room temperature condition.

7. Conclusion

The study has been carried out with the objective of understanding the behavior of Barmer bentonite under the influence of temperatures of 25°C, 60°C, 80°C, 100°C, and 120°C. The extensive experimental analysis shows that the liquid limit of Barmer bentonite decreases with increases in temperature. The initial value of the liquid limit at 25°C was 331 % and it decreased to 291 % when temperature reaches up to 120°C. There is a 12 % reduction in the value of the liquid limit with an increase in temperature. It has been observed that with an increase in temperature, the plastic limit decreases. At room temperature, the value of plastic limit 36 % and it decreased up to 32 %. The plastic limit of bentonite decreases by 11.11 % with a change in temperature. The plasticity index of Barmer bentonite also decreases with an increase in temperature. The maximum dry density of Barmer bentonite has been determined at different temperatures and it shows an increasing trend with temperature. The value of maximum dry density at room

temperature was 11.7 kN/m³ and as the temperature increased, it attained the value of 13.12 kN/m³. The change in maximum dry density is observed as a 10.2 % increase from an initial value.

It has been observed that there is a small variation in the liquid limit, plastic limit (decrease with temperature), and maximum dry density (increase with temperature) of Barmer bentonite with a change in the temperature. Such trends indicate that the properties of studied bentonite are favorable for its application as buffer material for nuclear waste repositories. Study of the behavior of bentonite at high temperatures has many applications. One of the important applications of bentonite is its use as an engineered barrier, and backfill material in case of deep underground repositories for safe disposal of high-level nuclear waste.

CRedit authorship contribution statement

Manish Kumar Jha: Writing – original draft, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Amit**

Jaiswal: Writing – original draft, Methodology, Investigation, Formal analysis, Data curation. **Amit Kumar Verma:** Writing – review & editing, Supervision, Conceptualization. **Trilok Nath Singh:** Writing – review & editing, Supervision.

Declaration of Competing Interest

The authors wish to confirm that there are no conflicts of interest associated with this publication and there has been no significant financial support for this work that could have influenced its outcome.

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