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Potential applications of marble dust in industrial use by characterization techniques – a review

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Abstract: Marble is a very common building material. In India, Rajasthan caters almost 85 % of the total demand of marble. It is normally a rich source of calcium carbonate which has various applications. Quarrying, processing and polishing of marble generates large amount of waste. This waste is dumped and left unattended thus creating hazardous problems. It is required to use this waste material in development of some value added products and reducing the impact on environment. Thus it is necessary to know the chemical properties of the waste left behind and how it reacts or helps in the hydration process. It is required to study the current uses of this waste marble dust in various industrial and construction practices. This paper describes various chemical properties of marble dust using characterization techniques like X-Ray Diffraction (XRD), X-Ray fluorescence (XRF), Thermo gravimetric Analysis (TGA) and Scanning Electron Microscope (SEM). Also the practical application of these characterization techniques in studying the chemical reactions taking place in mortars and concrete is reviewed. Suggestions for further studies and utilization in various fields for building a sustainable environment are also incorporated. It would encourage bulk utilization of waste marble dust as an alternative construction material which is otherwise a waste.

Keywords: *Marble Dust, XRD, XRF, SEM, TGA, Sustainable Environment*

Introduction:

Large amounts of solid wastes on large areas are produced by the marble processing industry. These are expected to increase as construction is continuously increasing as production of marble industry has been increasing annually in the recent years. Various natural stones like marble, slate granite etc which are cut and processed for used in industry are referred to as dimensional stones. Dimensional stones are characterized by aesthetics/acoustics and practicality in use. Marble is a crystalline, compact variety of metamorphosed limestone, consisting primarily of calcite ($CaCO_3$), dolomite (Ca Mg $(CO_3)_2$) or a combination of both minerals.

Two types of by-products are produced in marble processing. 30% of the stone during processing of the unprocessed stone goes to scrap because of being in smaller size and/or irregular shape.

It is basically the water containing marble powder which is left unused.

The water is reused till it gets thick enough (70% water – 30% marble powder) to be insoluble for marble powder. It can be approximately estimated that in processing of 1 tonne of marble produces equivalent amount of marble slurry.

The heaps of this waste material are not only hazardous to health but also acquire large land areas and remain scattered all around. Thus spoiling the aesthetics of the entire region and have affected the tourism and industrial potential of the places nearby the processing units. The dumping practices thus pose a severe threat to the environment, eco - system and health of the people.

This also contaminates the surface and underground water reserves The wastes of this industrial activity can reach even 20% wt of the raw marble (Fernandes et al., 2003). Apart from reducing the consumption of cement by a significant amount, it would be eco-friendly as it would make use of the industrial waste.

In today’s industrial concept waste material of one industry is normally used as a raw material for some other industry so that it could be utilized to its maximum capacity. It would help in -

(a) Reducing the environmental impact produced from marble manufacturing as waste and the management cost of waste produced.

(b) Producing suitable building materials that have their characteristics with regards to the adopted or active standards without any municipal risk.

(c) Maximizing the use of natural resources in industry.

Characterization techniques:

Advances in the science and technology of cements and admixtures are related to the utilization of different techniques and proper interpretation of the data derived from them. Therefore it is necessary to study and use the various techniques in order to acquire the data related to marble dust.

1 Thermo gravimetric analysis (TGA) is a thermal analysis method in which changes in physical and chemical properties of materials are found out. It helps in determination of weight changes that occur when a sample is heated at a uniform rate. This technique provides information on the thermal stability of a particular sample and composition of the intermediate and the residue. (Ramachandran, 1995)

Thermo-gravimetric analysis is a precise quantitative measurement of the weight change of a solid as it is heated at a controlled rate. TGA records change in mass from dehydration, decomposition and oxidation of a sample with time and temperature (Valeria et al., 2010)

Common applications of TGA are –

(1) Material characterization through analysis of characteristic decomposition patterns
(2) Study of degradation mechanisms and reaction kinetics,
(3) Determination of organic content in a sample,
(4) Determination of inorganic (e.g. ash) content in a sample, which may be useful for corroborating predicted material structures or simply used as a chemical analysis.

Most manufacturers in today's market no longer make true DTA systems but rather have incorporated this technology into thermo gravimetric analysis (TGA) systems, which provide both mass loss and thermal information. With today's advancements in software, even these instruments are being replaced by true TGA-DSC instruments that can provide the temperature and heat flow of the sample, simultaneously with mass loss. These instruments are normally called SDT (simultaneous DSC/TGA).

2 Differential thermal analysis (or **DTA**) is a thermo analytic technique which is similar to differential scanning calorimetry. In DTA, the heat effects associated with chemical or physical changes are recorded as a function of temperature or time, as the substance is heated at a uniform rate. (Ramachandran, 1995) This differential temperature is then plotted against time, or against temperature (DTA curve, or thermo gram). Changes in the sample, either exothermic or endothermic, can be detected relative to the inert reference.

(Valeria et al., 2010) carried out TGA Thermal analysis to show that the marble dust contains about 66% of calcium carbonate, $CaCO_3$. From Fig 1 it could be observed that there is a sharp weight loss (corresponding to the flex of the DTA curve) occurs from about 730 °C–900 °C while strong heat absorption

was detected. It is due to the decomposition reaction of calcite, which is endothermic.

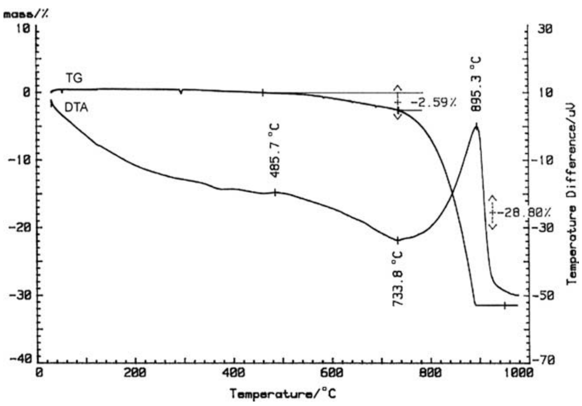

Figure 1: *Results of TGA and DTA of marble dust sample (Valeria et al., 2010)*

Bilgin et al (2011) used three different waste marble powders for use as a material in making industrial bricks. Waste marble powders were grinded under 40 lm before the characterization studies.

DTA (differential thermal analysis) studies to examine the thermal behavior of waste marble powders were carried out up to 1000 °C. In differential thermal analysis, the characteristic peak for Calcite mineral occurs between the temperatures of 850° - 900 °C. It can be easily understood from the diagram of BN3 in Fig 2 clearly describes that the calcite in the mineral decomposes at the range of 850° - 900 °C temperature in continuous heating and gives a strong endothermic peak.

Calcite, as a result of decomposition, turns into CaO in solid phase and CO_2 in gas phase. $CaCO_3$ minerals Calcite and Aragonite have the same chemical composition but different crystal systems. But in this material Aragonite were not present as they occur in the range of 420 – 485 °C in an endothermic reaction (Bionco et al., 2005)

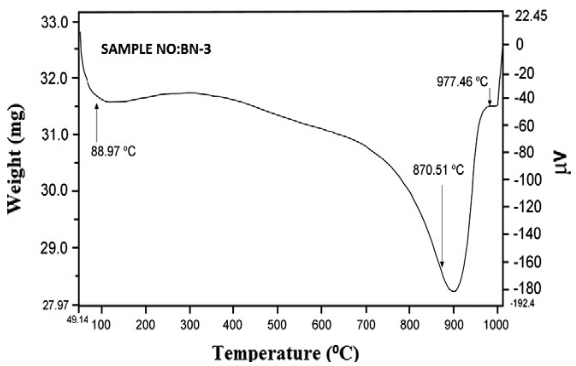

Figure 2: *TGA/DTA curve for marble powder sample BN3 (Bilgin et al., 2010)*

Aliabdo et al., (2014) used TGA to identify the reactions occurring in concrete for use of marble dust as a partial replacement of cement. In Fig 3 TGA profiles of cement paste samples with 0%, 7.5% and 15.0% marble dust as cement replacement are shown. The dehydration of calcium silicate hydrate (C–S–H) gel and ettringite is identified at a temperature below 200 °C for all specimens is shown on the TGA profiles.

Percentage of Weight losses was studied at different temperature ranges thus studying the decomposition of calcium hydroxide. For all specimens; the weight loss of calcium hydroxide was found in the temperature range 450–500 °C thus indicating that there was no change in chemistry phase. Decomposition of carbonate phases follows thermal decompositions of calcium hydroxide that is above 600 °C for all specimens.

It could be noticed that the use of marble dust does not affect the weight loss of calcium hydroxide dramatically which indicates that there is no change in chemistry phase. Thus it shows that there is no active role of marble dust in reactions in chemistry phase in concrete.

3 Scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons.

It helps in understanding of the microstructure of hydrated cementitious pastes. The technique consists of a narrow electron beam scanning the surface of a specimen. Due to the large depth of focus it enables to give a three dimensional arrangement.

Clear indications in the literature have been obtained of the presence of hexagonal crystals of $Ca(OH)_2$ and calcium monosulfate aluminatre hydrate.

Kavas and Olgun (2007) used SEM micrograph to study the mortar specimens cured for 28 days as shown in Fig 4 and 5. They found out from Figure 5 that abundant hydrated phases as well as $Ca(OH)_2$ crystal intermixed with calcium silicate hydrate existed in mortar.

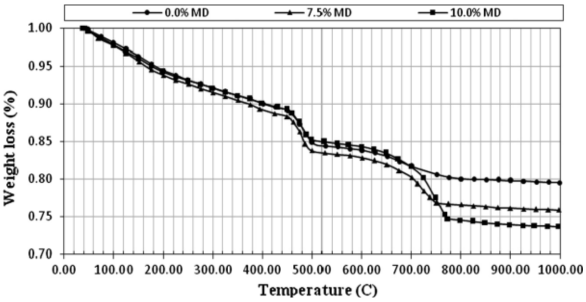

Figure 3: *TGA curves for paste samples (Aliabdo et al., 2014)*

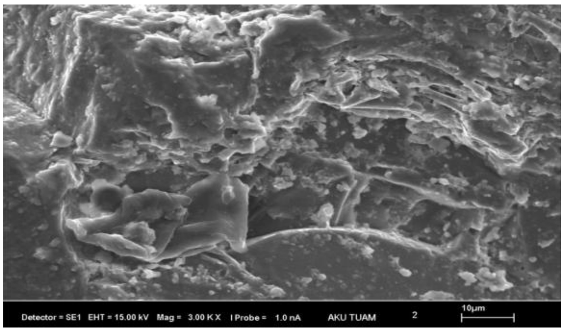

Figure 4: *SEM after 28 days of hydration (the cement used contains 65% PC ++ 5% G + 22% L + 8% MD). Kavas and Olgun (2007)*

Albado et al., (2014) A number of scanning electron microscope (SEM) micrographs that illustrate the micro-structure characteristics of some marble dust modified cement pastes at 28 days are shown in Fig. 6-8. During scanning process, it was observed that marble dust blended cement specimens are denser and less porous than control specimens. At this age, the microstructure of pastes is composed of amorphous particles of calcium silicate hydrate (C–S–H) and calcium hydroxide (CH) crystals that appear in massive layers. Ettringite (E) needles are located in pores.

4 X-Ray Diffraction XRD – This technique is capable of identifying, estimating and elucidating the structure of many unhydrated and hydrated phases of Portland cement. It cannot however be efficiently used for C-S-H and other phases which are extremely fine-grained and nearly amorphous but can be useful in determining the crystalline phases.

It can help in determining the kinetics of hydration of cement both in the presence and absence of admixtures. XRD can also help in correct determination of the specific surface are of cement pastes for different conditions of exposure to H_2O

Mineral phases are generally determined by comparing with the JCPDS index (JCPDS, 1985)..

Kavas and Olgun (2007) studied the XRD patterns of CB and MD bricks shown in Figures 9 and 10, respectively. XRD patterns of all brick showed that they were composed of quartz, being the main component, illite, albite, and hematite.

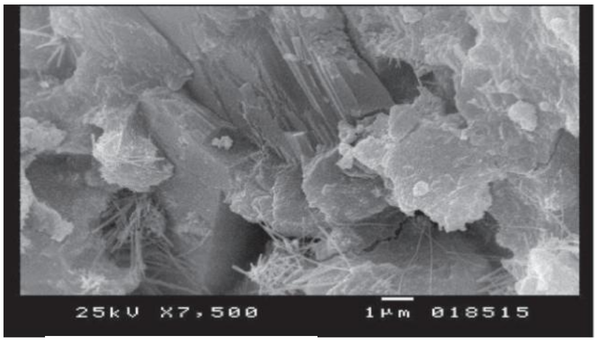

Figure 5: *Control paste (Aliabdo et al., 2014)*

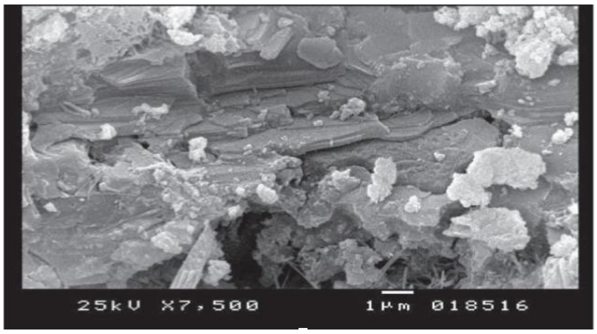

Figure 6: *7.5 % paste (Aliabdo et al., 2014)*

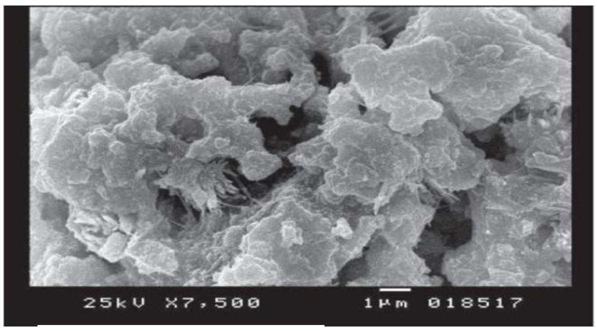

Figure 7: *10% MD paste (Aliabdo et al., 2014)*

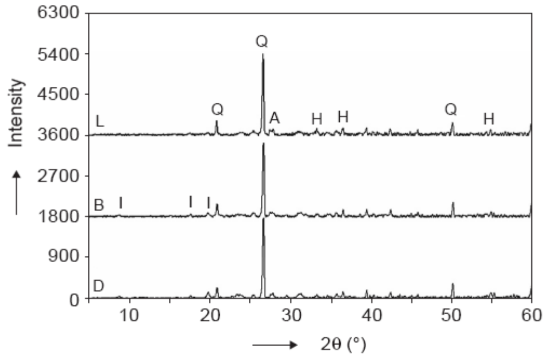

Figure 8: *XRD pattern of crushed brick. Q - Quartz; I - illite; A - albite; H - hematite. (Kavas and Olgun 2007)*

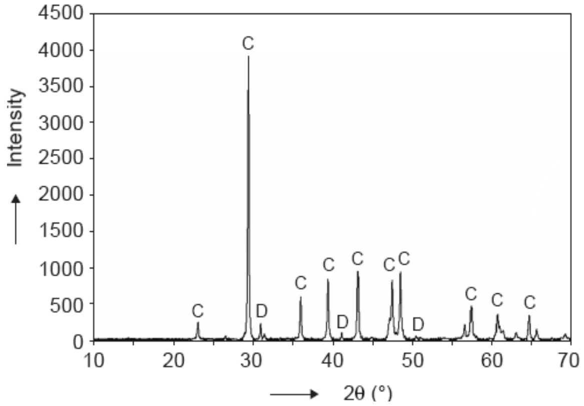

Figure 11. *XRD spectrum of the marble dust. C - Calcite; D -dolomite. (Kavas and Olgun 2007)*

(Valeria et al., 2010) used XRD to characterize marble powder. Fig. 11 shows the presence of quartz, which could be estimated at about 3%, and ankerite (ferroan dolomite) at about 2%. The remaining part of the marble powder consist of amorphous silica or silicates, coming from natural stones other than marble, whose low crystallinity (making them mostly undetectable by X-ray diffraction) may be due to mechanical processing (sawing and shaping).

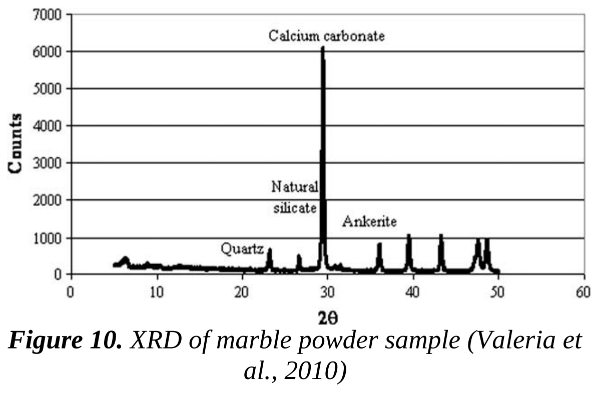

Figure 10. *XRD of marble powder sample (Valeria et al., 2010)*

Bilgin et al., (2011) used XRD results of marble dust sample to characterize it. It was observed in marble waste marble powders mainly present were Calcite (CaCO3) and small amount of Dolomite (MgCO3_CaCO3).

According to PDF (powder diffraction file) cards from the JCDPS index, the main peaks of Calcite were at 23.077 Å, 29.414 Å, 35.919 Å with the other minor peaks were determined. XRD in Fig 12 results clearly showed that marbles consist of mainly Calcite minerals.

X-ray fluorescence (XRF) – In this the sample is irradiated by an intense x-ray beam; the elements in the specimen emit their characteristic x-ray spectra. The wavelengths of the spectral lines may be used to identify the element and the intensity of the line determines the concentration of the element. This technique is different from the x-ray diffraction method in that the elements rather than the compounds are identified and estimated. Analysis of elements above atomic no.12 is accomplished easily. It is normally used for determining the chemical composition of powdered materials.

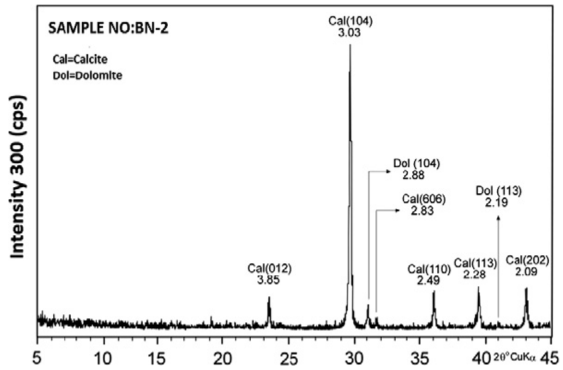

Figure 11: *XRD sample of BN2*

Ali Aliabdo (2014) used XRD to compare between hydration phases control paste (without marble dust) and pastes with marble dust blended cement. It indicated that there is no observed difference between the studied specimens, especially regarding to calcium hydroxide (CH) contents as seen in Fig 13. These results confirm the test results of TGA. Also, there is no difference in the intensity of the other components among control sample and samples made with marble dust blended cement.

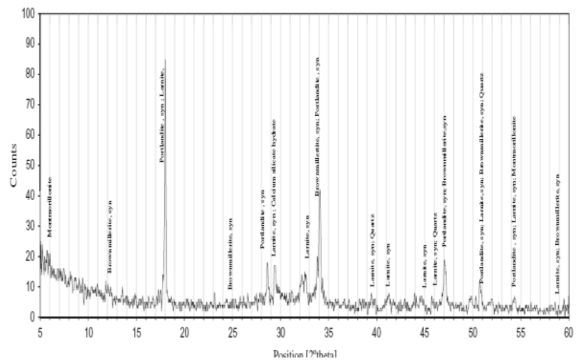

Figure 12: *XRD of control sample (Ali Aliabdo 2014)*

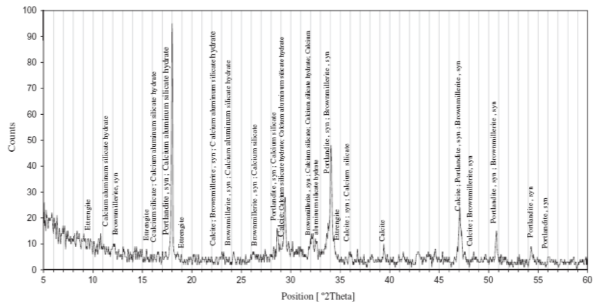

Figure 13: *XRD of sample with 7.5 % MD (Ali Aliabdo 2014)*

Various authors have used and found out the chemical composition of marble dust in the literature and they are stated in table 1.

2. Use of Marble Waste as a cement and sand replacement:

Sahu, *et al.* (2003) investigated the use of crusher dust as a partial replacement of fine aggregates in concrete while Rao, *et al.* (2004) as complete replacement of fine aggregates. Gupta et al. (2008) studied the use of partial replacement of cement by marble dust. A case study in the cost estimation analysis for the influence of Marble powder/granules in Concrete mix was performed in Nehru National Institute of Technology, Allahabad. Pitroda, *et al.* (2013) Compressive strength of the concrete has increased with increasing percentages of marble dust additions. Ali Aliabdo (2014), Gurumoorthy (2014) Shirulae et al (2014) and Hebhoub et al. (2011) also studied the effects of partial replacement of cement and sand by marble dust. By using the marble dust the rate of the concrete is decreasing and strength is increasing.

Due to marble dust, it proved to be very effective in assuring very good cohesiveness of mortar and concrete. From research studies, it is presented that the marble dust can be used as a replacement material for cement; and 10% replacement of marble dust gives an excellent result in strength aspect and quality aspect and it is better than the control concrete. The results showed that the substitution of 10% of the cement content by marble stone dust induced higher compressive strength, higher splitting tensile strength, and improvement of properties related to durability. Test results show that this industrial waste is capable of improving hardened concrete performance up to 15%, enhancing fresh concrete behavior and can be used in plain concrete. The marble slurry dusts not only for the RCC but also in case of pavement design have proven results.

3. Use of Marble Dust in Bricks construction:

The effect and usage of limestone as an additive in cement and concrete were investigated by several researchers in recent years. Kavas and Olgun (2007) used marble dust (MD) generated by the marble cutting industries and crushed bricks (CB) cement replacement materials. The properties of cement and mortar containing MD and CB were investigated. The setting time of the cements were retarded when the waste material replaced a part of the clinker. Waste marble is well usable instead of the usual aggregate in the paving block production (Gencela et al., 2012).

Huseyin et al. (2010) in Ankara, Turkey investigated the usability of waste marble dust (WMD) as an additive material in blended cement has been investigated. Obtained results showed 10% WMD can be used as an additive material in cement manufacturing. Bilgin (2011) investigated the usability of waste marble dust as an additive material in industrial brick production.

Aukor (2009) studied the possiblility of using marble sludge powders (MSPs) as a substitute for limestone was investigated over a three year period compressive strength 9 N/nm2 at 28 days, and water absorption (7%), anticipating a complimentary usage of sludge as a by-product instead of being waste

4. Use of Marble Dust in Asphaltic Concrete:

The use of marble sludge in mixture as filler material in asphalt manufacture was investigated by Puzinauskas (1983) and Terzi (2000).

During the shaping process of marble the marble dust collected can be used as an alternative filler material in asphalt mixtures (Karasahin and Terzi, 2004).

In order to evaluate the properties of each type of filler Huyesin (2007) and Adil N Abed and Sadoon O Eyada, conducted tests which consist of the grain size distribution, the specific gravity (Gs), specific surface area (SA), pore volume (PV), mineral composition, pH and chemical composition. Also to study the effect of marble dust on the performance of HMA mixture various tests like Los Angeles abrasion, aggregate impact value, freezing and thawing, flakiness index and Marshall Stability flow tests were carried out on the aggregate specimens.

1. The use of Marble waste as a filler in HMA concrete increases Marshall Stability of mix.
2. It also increases the air voids and flow, and decreases the density of mix.

Waste marble dust can be used as filler the results indicated that waste marble aggregates could be used in light to medium trafficked asphalt pavement binder courses in hot mix asphalt concrete moreover the following reasons:

1. Since it is a byproduct (waste) of cement productions, it is very cheap and has an economical advantage.
2. Its usage will decrease the side effect of pollution caused by the accumulations of tons of Waste in situ.

5. Use of Marble dust in Soils:

Pitroda, et al. (2009) has presented that marble slurry dusts can be gainfully utilized in bulk quantities in construction of road pavement layers and in embankments. Use of marble slurry dusts results in saving of soil and savings on difference in cost of natural material soil, besides protection of environment. Ismail Zorluer. (2010) studied the used marble dust as an additive material in clay soil. Arora and Ameta (2014) used marble powder for mechanical stabilization of cohesive soil. CBR and shear strengths tests were carried out. According to the results 20% addition in soil can improve the soil stability.

1. Marble slurry can be recommended for road and embankment construction.
2. It decreases the consolidation and swelling index of clayey soil.
3. Compression index (cc) and expansion index (ce) of specimens decreases when the amount of the added marble dust increases.
4. Void ratio decreases thus reducing consolidation

Conclusions and Recommendations:

Characterization techniques help us to identify the chemical compositions and physical properties of marble dust, thus helping to point out the potential areas where the raw material could be used and also in studying the reactions occurring in various processes.

1. The results of TGA analysis of marble dust show that it mainly comprises of $CaCO_3$ in the range of 850-900°C. It also helps in identifying the chemical changes occurring in the hydration phase of concrete with the help of studying weight losses by constantly heating the sample.
2. SEM micrographs help in determining the hydrated phases and the formation of calcium hydroxide in these phases.
3. With the help of XRD it could be inferred that marble dust mainly shows peaks of $CaCO_3$ thus matching with the results of TGA.
4. XRF helps in determining the chemical composition of the material thus helping in characterization of material as pozzolonic or non pozzolonic.

Feasible Uses of Marble Waste - The areas where the utilization of marble waste and marble slurry is being used and could be used as a substitute for conventional raw materials are as follows:

1. It can be used a filler material for roads and embankment in which water bound Macadam can be laid. The preliminary results have been very encouraging according to the literature
2. For manufacture of bricks Marble slurry as it is chemically dolomitic in nature and consists of very fine particles.
3. Using marble slurry as a partial replacement of cement or sand in conventional concrete as a filler material only because according to the results it does not take part in the hydration process.
4. Adding Marble waste to the HMA mix reflects a good effect on the cohesion of mix due to high values of Indirect Tensile Strength While showing high effectiveness to change in temperature with high value of Temperature Susceptibility. Thus it could be used a filler in HMA.
4. Waste marble slurry could be used as a raw material for production of Ceramic Wall tiles needs to be evaluated on a pilot plant level.
5. Manufacture of lime Limestone is the main raw material for the production of Lime. Limestone can be replaced by marble waste.
6. Hollow Blocks and Wall Tiles Marble slurry waste and other clay products can be used in the production of Hollow prefabricated blocks for buildings if used in the right proportion.

It is also being used -

In waste water treatment, for de-sulfurising stack gases from utility and industrial plants that operate coal burning boilers, in the treatment of Sewage sludge to quell obnoxious fume, in filter beds as a screened mineral aggregate

The marble waste and marble slurry can be used as a substitute for limestone in various industrial applications.

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Table 1: *XRF values from literature.*

	LOI	*Silica*	*Alumina*	*Lime*	*Magnesia*	*Iron Oxide*	*Soda*	*Potash*	*SO3*	*TiO2*
Huseyin et al. (2010)	43.4	0.67	0.12	54.43	0.59	0.08	0.14	NA	NA	NA
Hebhoub et al. (2011)	44.26	0.15	0.08	54.86	1.03	0.04	NA	NA	NA	NA
Demirel(2010)		28.35	0.45	40.45	16.25	9.7				
Marras et al (2010)	42.62	0.43	0.14	54.07	0.6	0.05				
Aliabdo el (2014)	2.5	1.12	0.73	83.22	0.52	0.05	0.96	0.27	0.56	
Rodrigues et al (2015)	2.8	18.95	4.83	63.61	1.54	3.53	0	0.6	3.25	