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Microwave Synthesis of ZrO_2 Nanoparticles and its Reinforcement in Geo-Polymer gel

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Abstract : The term "**Nanotechnology**" was first defined by **Norio Taniguchi** of the Tokyo Science University in 1974. Nanotechnology, shortened to "**Nanotech**", is the study of manipulating matter on an atomic and molecular scale. Generally nanotechnology deals with structures sized between 1 to 100 nm and involves developing materials or devices within that size. For comparison, 10 nanometers is 1000 times smaller than the diameter of a human hair. Nanotechnology hasthe capacity to improve our ability to prevent, detect, and remove environmental contaminants in air, water, and soil in a cost effective and environmentally friendly manner. Nanoscience and nanotechnologies are revolutionizing our understanding of matter and are likely to have profound implications for all sectors. The present work involves to study the Microwave assisted synthesis and characteristic properties of Zirconium oxide (ZrO_2)nano powder and its reinforcement in geopolymer gel. Nanopowder was prepared by the citrate sol-gel method. The characteristic properties of nanoparticles are studied by the FTIR, UV-Visible, and XRD analysis, results the presence of tetragonal-ZrO_2nanopowder with particle size of 24.24nm. The geopolymer paste was prepared by mixing of fly ash with an alkali silicate solution in a solid to liquid ratio of 1. The ZrO_2nanopowder was mixed with geopolymer in the ratio of 0.5:5grams, and poured in to moulds to prepare nanoparticle reinforced geopolymer. The characteristic properties of geopolymer are stidied by the Compressive strength, Thermal resistivity and SEM analysis. It is found that small amount of ZrO_2nanopowderwill increases the compressive strength of geopolymer with its NaOH molarity increment, and also shows the high thermal resistivity at 300°C temperature.
Key words : ZrO_2nanopowder, Fly ash, Geopolymer gel, Compressive strength, Thermal resistivity.

Introduction

Nano materials are defined as engineered materials with a least one dimension in the range of 1-100nm. Particles of "**nano**" size have been shown to exhibit enhanced or novel properties including reactivity, greater sensing capability and increased mechanical strength. The nano technique offers simple, clean, fast, efficient, and economic for the synthesis of a variety of organic molecules, have provided the momentum for many chemists to switchfrom traditional method.Nanotechnology involves the study of the synthesis, characterization and Properties of nano materials, which have at least one characteristic dimension that is less than 100 nm. The application of nanomaterials can be historically traced back to even before the generationof modern science and technology. In 1857,**Michael Faraday** published a paper which explained how metal nano particles affect the colour of church windows. In 1959, **Richard Feynman** (awarded Nobel prizeinPhysics in 1965) gave a lecture titled "**There'sPlentyof Room at the Bottom**", suggesting the possibilityof manipulating things at atomic level. He speculatedon the possibility and potential of nanosized materials. This is generally considered to be the foreseeingof nanotechnology.Nanoparticle research is currently an area of intense scientific interest due to a wide variety of potential applications in biomedical, optical and electronic fields.In the presence of chemical agent[1-5]. Metal oxides play a very important role in many areas of chemistry, physics and materials science. Metal Oxide nanoparticles can exhibit unique physical and chemical properties due to their limited size and a high density of corner or edge surface site[3].

Zirconium dioxide is one of the most studied ceramic materials. ZrO_2 adopts a monocliniccrystal structure at room temperature and transitions to tetragonal and cubic at higher temperatures. Zirconium dioxide (ZrO_2) has a wealth of potential applications as High temperature and corrosion resisting components, bearing nozzle, smelting crucible. The geopolymers depend on thermally activated natural materials like Meta kaolinite or industrial byproducts like fly ash or slag to provide a source of silicon (Si) and aluminum (Al). These Silicon and Aluminum is dissolved in an alkaline activating solution and subsequently polymerizes into molecular chains and become the binder[2].

Results and Discussions

In the present work Zirconium oxide (ZrO_2) nanopowder was prepared by the microwave assisted process by Citrate Sol-gel method by using of Zirconium oxychloride and anhydrous citric acid as precursors. Geopolymer paste was prepared by mixing of fly ash with an alkali silicate solution by solid to liquid ratio of 1. The characteristic properties of ZrO_2nanopowder and its reinforcement in geopolymer are studied by the FTIR, UV-Visible, XRD, SEM, Compressive strength and Thermal resistivity techniques as follows.

FTIR-ATR Spectra of ZrO_2Nanoparticle

The Attenuated total reflectance (ATR) spectral method is used to analyze the Zirconium dioxide nanoparticles IR region, as shown in the following spectrum1.1

FTIR-ATR Analysis

The FTIR Spectra of zirconia nanoparticle is measured between the regions of 4000-400 cm^{-1}. The nanoparticles are shows various absorption peak values as follows. The peak value obtained at the region of 481.98 cm^{-1} is indicates Zr-O vibration band that shows presence of ZrO_2structure.The absorption peak is obtained at 1392 cm^{-1} is indicates presence of O-H bond. The Peak value obtained at the region of 1534 cm^{-1} is indicates absorption moisture. The peak obtained at the region above 3000 cm-1 is indicates stretching of 0-H bond, that shows presence of highly hydrated compounds[1].

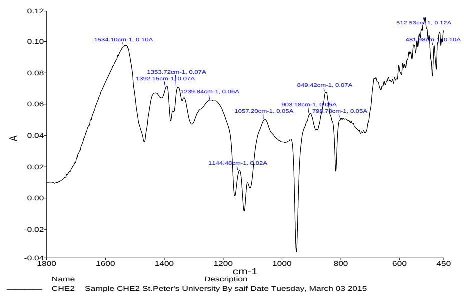


Figure 1: FTIR-ATR Method

The most significance of the FT-IR spectra of ZrO_2 nanoparticles are recorded in the region of 4000-400cm^{-1}

UV-Visible Analysis

The UV-VISIBLE-DRS Spectra of zirconia nanoparticle is measured between the regions of 200-1100 nm. The nanoparticles show the sigle sharp absorption peak value as follows. A single absorption peak value obtained at 347nm, is indicates the presence of tetragonal ZrO_2 nanoparticles[1].

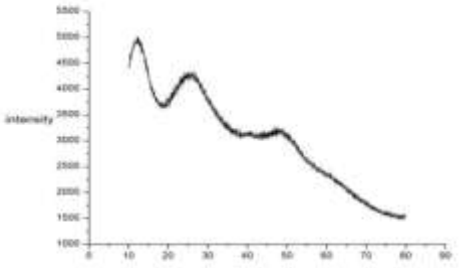
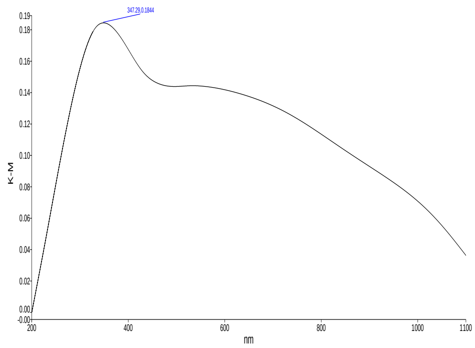


Figure 2: UV-VISIBLE-DRS Method

The UV-VISIBLE-DRS spectra of ZrO_2 nanoparticles are recorded in the region of 200-1100 nm.



SEM image of ZrO_2 nanoparticle mixed Geopolymer gel.

The scanning electron microscopic image shows that the ZrO_2nanoparticles are evently spread over the geopolymer Composite and size of the nanoparticle is around 24 nm. shown in the Fig.3.

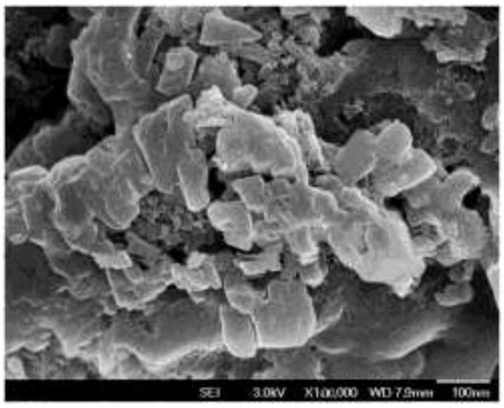


Fig.3 : SEM image of ZrO_2 nanoparticle

XRD- Spectra of ZrO_2 Nanoparticle

X-ray powder diffraction (XRD) spectral method is used to find the size and phase of the Zirconium dioxide nanoparticles, as shown in the following spectrum. Zirconia nanoparticles were synthesized by the Citrate sol-gel method, and calcinated at400C temperature up to 6 hrs. These nanoparticles were characterized by XRD technique. nanoparticles have an approximately Rectangular shape with an average size of 24.24 nm.

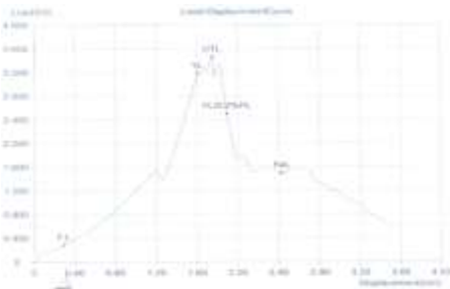
Hence they shows tetragonal zirconia nanoparticles phase which have the high surface area, shows a good catalytic activity is shown in the following spectrum 1.3[4].

Spectrum1.3: XRD – Spectrum of ZrO_2 Nanoparticles

XRD-Spectra of ZrO_2 nanoparticle powder was shown in the following spectrum.

Compressive strength of ZrO_2Nanoparticle reinforced Geopolymer moulds

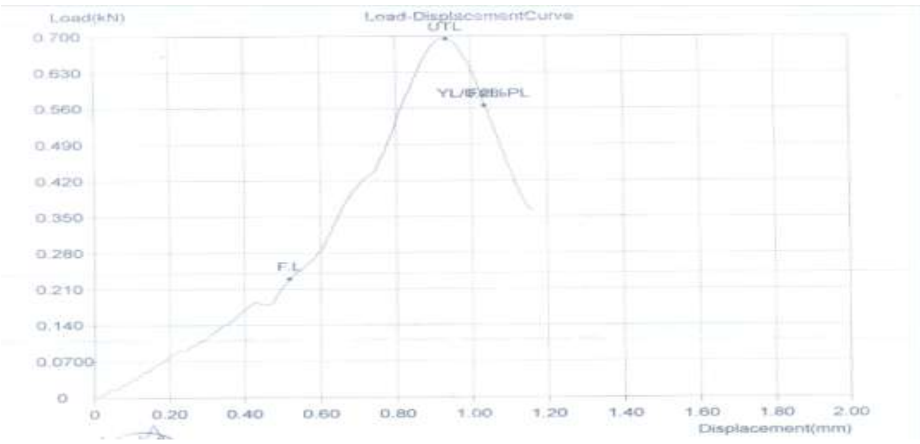
Mould-1: Mould-1 had prepared by the mixing of (10 molar NaOH solution=15mL +Sodium silicate solution=15ml) alkali activated solution= 20mL + fly ash20gm + 2gm of ZrO_2 nanoparticle powder. Is shown in the following graf.1.1



Graf1.1: compressive strength of Mould-1

The Ultimate compressive strength of mould was measured as 6.811Mpa

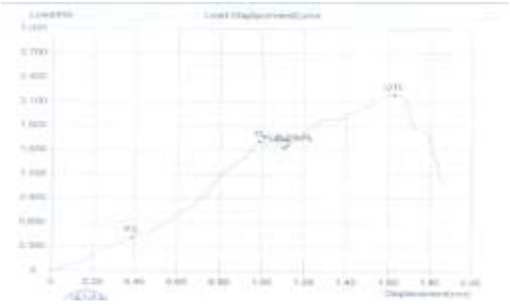
Mould-2: Mould-1 had prepared by the mixing of (7 molar NaOH solution= 7mL +Sodium silicate solution= 7ml) alkali activated solution= 15mL + fly ash 15gm + 1.5gm of ZrO_2 nanoparticle powder.Is shown in the following graf.1.2



Graf1.2: compressive strength of Mould-2

The Ultimate compressive strength of mould was measured as 2.075Mpa

Mould-3: Mould-1 had prepared by the mixing of (7 molar NaOH solution= 7mL +Sodium silicate solution= 7ml) alkali activated solution= 15mL + fly ash 15gm + 1.5gm of ZrO_2 nanoparticle powder.Is shown in the following graf.1.3.



Graf1.3: compressive strength of Mould-3

The Ultimate compressive strength of mould was measured as 4.22Mpa.

Table-1 Ultimate compressive Strength and Strain Measurements

S.No	Specimen With Differet Molariy Of NaOH	Size Of The Specimen	Ultimate Compressive Strength	Ultimate Displacemet (MPA)	Ultimate Compessie Strain (%)
1	10Molar	D= 25.45mm H = 41mm	6.811	1.8mm	4.39%
2	7Molar	D=20.65mm H=40mm	2.075	0.96mm	2.4%
3	7Molar	D=25.98mm H=24mm	4.112	1.62mm	6.75%

Compressive strain measurements:

1. 1.8/41 x 100 = 4.39% (Mould-1)
2. 0.96/40 x 100 = 2.4% (Mould-2)
3. 1.6/24 x 100 = 6.75% (Mould-3)

The geopolymer prepared by the mixing of ZrO_2nanoparticle shows various compressive strength depends on the size and diameter of the mould.

- The Mould-1 shows highest compressive strength = 6.811Mpa.
- The Mould-3 shows the highest compressive strain (%) = 6.75%.

Thermal resistivity

The ZrO_2 nanoparticles shows high thermal resistive character, In the present work these nanoparticles are used as non alumino-silicate binding materials for to prepare the geopolymer moulds by various length and diameters, these prepared moulds are left for dry up to 7 days, after that period moulds are heated at 300°C temperature up to 6hrs, which is shown in the following figure 4.9. The Geopolyme rmoulds are heated about 300°C temperature up to 6 hoarse, there is no any considerable changes are occurs in the moulds Hence it had concluded that Zirconium dioxide nanoparticles reinforce Gepolymer moulds are shows high thermal resistivity at 300°C temperature.

Conclusion

It has been concluded that the small amount of ZrO_2 nanoparticles will increase the binding capacity in fly ash geopolymer gel, and affects the chemical and physical properties of a geopolymer matrix.In the present work the Rare earth metal-oxide nanoparticle of ZrO_2 can be used as non-alumino silicate binding material in the fly ash based geopolymers.The small amount of ZrO_2 nanoparticle will increase the binding strength, these ZrO_2 nanoparticles reinforced geopolymer showed the high thermal resistivity up to 300°C.The 25mm diameter and 40mm height of ZrO_2 nanoparticles reinforced geopolymer mould-1 showed the high compressive strength of 6.811Mpa (Mega Pascal)The 25.98mm diameter and 24mm height of ZrO_2 nanoparticles reinforced geopolymer mould-3 showed the high compressive strain of 6.75%.

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