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Author post-print (accepted) deposited by Coventry University's Repository

Original citation & hyperlink:

Millogo, Y, Morel, JC, Traoré, K & Ouedraogo, R 2012, 'Microstructure, geotechnical and mechanical characteristics of quicklime-lateritic gravels mixtures used in road construction' *Construction and Building Materials*, vol 26, no. 1, pp. 663-669

<https://dx.doi.org/10.1016/j.conbuildmat.2011.06.069>

DOI 10.1016/j.conbuildmat.2011.06.069

ISSN 0950-0618

ESSN 1879-0526

Publisher: Elsevier

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Microstructure, Geotechnical and Mechanical characteristics of quicklime-lateritic gravels mixtures used in road construction

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Abstract

Microstructure of quicklime-lateritic gravels mixes, containing up to 8 wt. % quicklime, was investigated by using X-ray diffraction, infrared spectrometry, differential thermal analysis, scanning electron microscopy and energy dispersive spectrometry. Geotechnical and mechanical properties of the mixtures were also measured. Quicklime additions resulted of reduction of clayey fraction, plasticity index, methylene blue value and maximum dry density and the increasing of optimum moisture content. The results showed also that quicklime admixtures resulted in the formation of calcium silicate hydrate type I (C-S-H (I)), portlandite and calcite. This CSH (I) was formed through the pozzolanic reaction between a part of the kaolinite and hydrated lime in the basic medium. Moreover, C-S-H (I) marked crystallization with curing time and observed for the quicklime amounts (≤ 3 wt. %), contributed to the improvement of mechanical properties. These lime modified lateritic gravels are convenient for base course in road geotechnical applications.

Keywords: Lateritic gravels, quicklime mixes, Microstructure, Geotechnical and Mechanical Properties, Base course, Burkina Faso.

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1. Introduction

Lateritic soils are widespread materials in tropical and subtropical regions. They are required for road construction. However, road sections built with raw lateritic soils subjected to deterioration mainly because of their poor geotechnical and mechanical properties but also with traffic intensification. For the Burkina Faso, an enclosed country, the road transport is very important for commercial activities throughout the country and with the neighbouring countries. For this purpose, it is important to have good roads. It is known that the natural lateritic gravels are not suitable to build resistant and durable roads, it is therefore necessary to improve such raw material.

To improve the latter properties, additions of lime, cement, fly ash, burnt clay-lime binder, silica fume and vegetable fibres were tested [1-8]. Physical and mechanical characteristics of lime amended clayey lateritic soils as well as the effects of lime additions on mineralogical changes of this raw material have been evaluated [2, 3, 7]. However a little attention has been paid to the investigation of the correlation between microstructure using scanning electronic microscopy coupled to the energy dispersive spectrometry analysis (SEM-EDS) and physical and mechanical properties on such composite material.

Our work was dedicated to the correlative study between the microstructure and the geotechnical and mechanical properties of quicklime modified lateritic gravels from Burkina Faso.

Discussions concerning the CSH formation mechanism and its type in this particular case, which is already debated in the literature for raw clay materials-lime mixtures, and the prospective use of the modified lime-lateritic gravels, have been carried on.

2. Materials and experimental procedures

2. 1. Materials

The mineralogical and chemical compositions of the concerned lateritic gravels, from Sapouy (Burkina Faso), are given in Table 1a and b. Values of some geotechnical and mechanical properties of this material are gathered in Table 2. According to Millogo et al. [9], the studied sample was suitable for sub-base course in road construction.

The quicklime chosen was commercial quicklime from Spain commonly used in Burkina Faso. This quicklime was crushed until 0.315mm as particle size. Its X-ray diffraction pattern showed that it contained calcium oxide (CaO), portlandite (Ca (OH)₂) and a small amount of magnesium carbonate hydrate (MgCO₃.H₂O) [10].

2.2 . Experimental procedures

The studied mixtures consisted of lime (up to 8 wt. %) and lateritic gravels. For samples preparation, the above mentioned materials were oven-dried at 105°C for 24 h, manually mixed, to prevent from any grain size change, and stored in hermetic plastic bags in order to avoid moisture contamination.

The plasticity index (PI) and the methylene blue value (MBV) of the mixtures were assessed according to the NF P94-051 [11] and NF EN 933-9 standards [12], respectively.

The optimum moisture content (OMC) and the maximum dry density (MDD), and the California Bearing Ratio (CBR) of mixtures were determined in accordance with the NF P94-093 [13] and NF P94-078 [14] standards, respectively. For the CBR measurements, differently treated samples were tested with a universal press (SEDITECH apparatus).

The mechanical tests were realized on cylindrical mixture samples which were compacted in CBR moulds at the optimum modified Proctor and kept in room at 20°C for 7, 14, 28 and 90 days. Tests were performed with a universal press and carried out according to the NF P 18-406 [15] standard for the compressive strength and the NF P18-408 [16] standard for the tensile strength.

The nature of crystalline phases in the prepared samples was identified using a Philips X'Pert MPD diffractometer equipped with a copper $K\alpha$ radiation ($K\alpha = 1.5418 \text{ \AA}$).

The infrared examinations were done on mixtures of 1mg of lime-lateritic gravels samples and 150 mg of KBr. They were performed using a Nicolet 510FT-IR spectrometer operating in the range $4000\text{-}400 \text{ cm}^{-1}$.

The thermal changes of the mixtures were identified using a Labsys-Setaram apparatus functioning at a heating rate of $10 \text{ }^\circ\text{C}/\text{min}$ and under N_2 atmosphere.

Microscopic examinations were done on carbon-coated pieces taken from the above-mentioned cylindrical samples using a Jeol JSM 5500 scanning electron microscope equipped with a Falcon EDAX analyser. The elemental quantitative analyses were performed without standards (ZAF method). The maximum resolution was around 3.5 nm.

3. Results and discussion

3.1. Mineralogical and microstructural characterization (SEM-EDS)

The X-ray diffraction patterns of powdered samples of lime-lateritic gravels (Fig.1) showed the presence of kaolinite, quartz, goethite, hematite, rankinite as well as calcite and portlandite. These two latter compounds were formed in the lime-lateritic gravels mixtures and their amounts increased with lime additions. The presence of kaolinite in the lime-lateritic gravels showed that only a fraction of this one had reacted with lime.

To highlight the potential presence of amorphous compounds, mixtures of lime-lateritic gravels were subjected to thermal and IR spectroscopy analyses. Regarding the thermal results (Fig. 2), the endothermic effects at about 80, 350, 520 and 567°C were linked to dehydration water, loss of crystallization water of goethite, dehydroxylation of kaolinite and to the transformation of quartz α to β , respectively. The exothermic effect at about 320°C expressed the combustion of organic matters. The weak thermal effect at 120°C was due to the deshydroxylation of calcium silicate hydrate (CSH) [7; 17-19], when the one at 472°C was attributable to the deshydroxylation of portlandite. The marked endothermic phenomenon at about 750°C was linked to the transformation of calcite to lime [18].

Concerning the infra-red investigations (Figs.3a-c), the spectra displayed bands related to quartz (780cm^{-1}), kaolinite: $3690, 3620, 3610\text{ cm}^{-1}$ (O-H stretching vibrations); $1050, 1030, 1010\text{ cm}^{-1}$ (Si-O vibration); 910 cm^{-1} (Al-OH bending vibration); 690 cm^{-1} (Si-O vibration) [20-22], and hygroscopic water (1640 cm^{-1}). The absence of band at 3672 cm^{-1} proved that the kaolinite of the sample had a low cristallinity. Bands associated with calcite ($2900, 1430, 874\text{ cm}^{-1}$) [13] and carbon dioxide (2350 cm^{-1}) were also present. This carbon dioxide was dissolved in lime-rich samples. The intensities of bands related to calcite and carbon dioxide increased with lime additions. It was worth noting that the marked intensification of the broad band at 3400 cm^{-1} , which was commonly attributed to hydration water, may be taken as an indication of the CSH presence [10, 23, 24]. The reduction of the relative intensities of O-H stretching vibrations bands of the kaolinite located at about 3690 and 3620 cm^{-1} (Fig.4) once confirmed the formation of CSH. . The maximum quantity estimated of kaolinite implied in the pozzolanic reaction with lime was 25wt.%.

SEM examinations and EDS analyses performed on raw material showed heterogenous structure consisting of isolated particles of fine and coarse euhedral grains of quartz (Fig.5a) and isolated and irregular particle of kaolinite (Fig.5b). With 2wt.% lime

sample, the texture was homogeneous with development of many fibrous compounds which joined the isolated particles (Fig.5c). This fibrous compound was the amorph CSH type I. According to EDS analysis of the above sample which showed the decreasing of the silicium peak of kaolinite and the presence of calcium peak, the CSH provided the pozzolanic reaction between portlandite and kaolinite minerals at the edges (tetrahedral layers for this case) in basic medium.

The proposed mechanism of CSH formation was summarised as:

- (1) hydration of lime to produce portlandite : $\text{CaO} + \text{H}_2\text{O} \rightarrow \text{Ca}(\text{OH})_2$;
- (2) dissociation of portlandite : $\text{Ca}(\text{OH})_2 \leftrightarrow \text{Ca}^{2+} + 2\text{OH}^-$;
- (3) formation of CSH after solubilisation of kaolinite silica:



The coexistence of kaolinite and CSH in the mixture showed again that only a part of the kaolinite reacted with lime. The 3wt. %lime sample presented a homogeneous microstructure. Almost all the isolated particles were linked with the cimentitious compounds (CSH) (Fig.5d). Calcite and/or portlandite were present taking into account the important intensity of calcium peak according to the analysis by EDS. As the lime content increased to 8wt. %, portlandite and calcite which were extensively formed and manifested as bright areas (Fig.5e). CSH was not well identified. This was confirmed by a slightly increasing of the relative intensities of O-H stretching vibrations bands of the kaolinite located at about 3690 and 3620 cm^{-1} . The excessive carbonation resulted of the occurrence of heterogeneous microstructure. The portlandite was formed by hydration of lime whereas the calcite provided the reaction between portlandite and carbon dioxide of air atmosphere according to the following equation: $\text{Ca}(\text{OH})_2 + \text{CO}_2 \rightarrow \text{CaCO}_3 + \text{H}_2\text{O}$.

The constancy of iron peak intensity proved that hematite and goethite were not implied to any reaction with lime additions. This result was similar to this reported by Bilong et al. [7].

It could be concluded that for lateritic and clayey soils, there are the same minerals which react with lime. These two types of soils chemically have the same behaviour during their lime treatment lime.

3.2. Geotechnical and Mechanical Properties

The grain size distribution obtained by humid sieving and sedimentation analysis for silt and clay was given in Fig. 6. The clay amount following the geotechnical definition ($<2\mu\text{m}$) was 5wt.%. The increase of grain size, in case of quicklime addition, was essentially due to the flocculation and agglomeration phenomena which followed the cation exchange reaction between Ca^{2+} and the cations of sample. The plasticity index (PI) and the methylene blue value (MBV) versus lime content are given in Fig.7. In general, the plasticity index and the methylene blue value decreased. For quicklime content $>3\text{wt.}\%$, the plasticity index increased. The decrease of the plasticity was also the result of exchange reaction which flocculated the sample particles reducing the clay-size fraction. This phenomenon also explained the decreasing of MBV values. The plasticity increase for this sample if the quicklime content $>3\text{wt.}\%$ was due to the excess of hydrated lime, more plastic than the sample which was not implied in any reaction. The reduction of methylene blue value was due to the reduction of clay fraction with the pozzolanic reaction between the clay fraction and the hydrated lime. All these phenomena were favoured with pH increasing (Fig.8). The small values of MBV and the presence of kaolinite in the different composites give the third evidence that the kaolinite is partially reactive with quicklime.

The optimum moisture content (OMC) and the maximum dry density (MDD) versus quicklime content are presented in Fig.9. The OMC increased while the MDD decreased with quicklime additions. The increasing of OMC was due to the high affinity of lime with water to

induce the dissociation of portlandite to produce Ca^{2+} , responsible for the cation exchange reaction.

The MDD behaviour has been studied in the case of compacted earth block (CEB) for building by Venkatarama Reddy and Jagadish 1993 [25], Mesbah et al. 1999 [26]. According to them the main cause of the decreasing of the MDD with water content is due to the increasing of water content of the soil. After drying, the water left the voids and that decreased the MDD. The reduction of MDD was also partly due to the replacement of some particles of the raw sample, more dense (with 2.74 as specific gravity) by quicklime (with 2.08 as specific gravity), less dense. The decrease was approximately 0.04g/cm^3 . It could be also due to the agglomeration of clay particles occupying a great volume while the sample mass increased slightly.

The results concerning the evolution of PI, OMC and MDD were similar to those obtained by Attoh-Okine (1990, 1995) [2, 3] and Osula (1996) [27] on the stabilization of laterite from Ghana and Nigeria respectively by lime up 3wt. The reduction of clay fraction with lime increase was accentuated with the laterites studied by Attoh-Okine and Osula than this study because of their higher clay fraction, which increased the flocculation and agglomeration phenomena. In fact the laterites of Ghana and Nigeria (forest laterites) had a clay fraction (17-22wt. %) and 13wt. % respectively than the one of Burkina Faso (savannah laterite).

The evolutions of CBR values after four days of immersion and three days drying in the air followed to four days of immersion, compressive and tensile strengths with quicklime contents were respectively plotted in Figs.10, 11 and 12. These curves showed the increasing of all these parameters with lime additions. The optimum values were obtained with 3wt. % quicklime. The increasing of these parameters was due to the formation of CSH during the pozzolanic reaction between kaolinite and quicklime. This cementitious compound linked the

isolated particles of soil reducing the micro-porosity as shown the SEM micrographs. The CBR values after three days drying in air following four days of immersion for the specimens were higher than those obtained after four days of immersion. This is mainly due to the increasing suction that increased the strength of the sample. This could be also explained by the fact that the pozzolanic reaction between quicklime and kaolinite was accelerated with air temperature (around 35°C during experimental period) [19, 28, 29].

The decrease of these parameters for quicklime content > 3wt. % was the result of the excessive formation of calcite and portlandite as shown by DRX, ATD, IR spectrometry and SEM studies. The calcite limited the formation of CSH and had a negative effect on the mechanical properties. The compressive and tensile strengths increased with curing time (Fig. 11 and 12). Between 7 and 14 days, these above mechanical properties significantly increased. This proved that the pozzolanic reaction was time dependent and was accelerated for the mixture of lime-laterite at early age.

The mechanical properties (CBR, compressive and tensile strengths) of the studied mixtures were better than those reported by Attoh-Okine (1990) [2]. The difference could be linked to the mineralogical composition of the samples. The studied lateritic gravels were less rich in clay minerals comparing to those studied by Attoh-Okine.

CBR values for the specimens dried in the air for three days and immersed for four days were higher than 80%. All these composites were suitable for base course in road construction according to the required standard [30, 31].

4. Conclusions

Based on the results obtained in this paper, the following conclusions were raised:

1. Lime additions generated the formation of new mineral phases such as calcite, portlandite and poorly crystallized calcium silicate hydrate type I (CSH (I)). These new mineral phases induced a homogeneous microstructure of the different composite. This CSH was formed through the pozzolanic reaction between a part of kaolinite and lime. Goethite and hematite did not react with lime therefore lateritic gravels and clayey material have the same behaviour with lime additions.
2. The plasticity index (PI), methylene blue value (MBV), maximum dry density (MDD) decreased with quicklime additions while the optimum moisture content (OMC) increased.
3. The mechanical properties (Californian bearing ratio, compressive and tensile strengths) increased with quicklime contents up 3wt. % and decreased for quicklime contents > 3wt. %. The increasing of these parameters was linked to the formation of CSH (I) and the minor formation of calcite and/or portlandite. The excessive formation of calcite and/or portlandite inhibited CSH formation and consequently the mechanical properties decreased.
4. The compressive and tensile strengths increased with curing time essentially due to the marked crystallization of CSH during the time. These mechanical strengths were higher than clayey sample amended with lime. This difference was linked to the grain size distribution. In fact, lateritic gravels were rich in gravel and sand which contribute to increase the mechanical properties.

5. The modified lateritic gravels with small amount of lime ($\leq 3\text{wt.}\%$) were convenient for base course in road construction and present an economic advantage for developing countries such as Burkina Faso.

Acknowledgements

The authors wish to acknowledge the financial support of “Aire- Sud Project” and “Laboratoire National du Bâtiment et des Travaux Publics” of Burkina Faso. This last laboratory gave its constant help for geotechnical and mechanical tests. Younoussa MILLOGO is grateful to Mr Kalsibiri KABORE in the “Laboratoire National du Bâtiment et des Travaux Publics” for his constant assistance for the realizations of geotechnical and mechanical tests. The authors wish also to acknowledge the assistance of Pierre-Antoine CHABRIAC for his contribution in the English correction of this paper.

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Table1-a**Chemical composition of the raw material**

| Oxides | SiO ₂ | Al ₂ O ₃ | Fe ₂ O ₃ | MgO | CaO | CuO | MnO | Na ₂ O | K ₂ O | TiO ₂ | L.O.I.* | Total |
|---------------|------------------|--------------------------------|--------------------------------|------|------|------|------|-------------------|------------------|------------------|---------|--------------|
| Wt.% | 56.19 | 10.1 | 16.09 | 0.05 | 3.24 | 0.11 | 1.68 | 2.01 | 0.07 | 1.39 | 8.14 | 99.07 |

*Loss on ignition

Table 1-b**Mineralogical composition of the concerned raw material**

| Minerals | Quartz | Kaolinite | Rankinite | Goethite | Hematite | Total |
|-----------------|--------|-----------|-----------|----------|----------|--------------|
| Wt.% | 42 | 26 | 6 | 15 | 3 | 92 |

Table 2**Some Geotechnical and Mechanical Characteristics of the raw material**

| Characteristics | Results |
|-------------------------------------|----------------|
| Atterberg Limits | |
| Liquid limit , w_L (%) | 22.5 |
| Plasticity limit , w_P (%) | 12.0 |
| Plasticity Index, PI (%) | 10.5 |
| Methylene Blue Value | |
| in (g/100g) | 0.17 |
| Modified Proctor | |
| Optimum moisture content (OMC), % | 6.6 |
| Maximum dry density (MDD), g/cm^3 | 2.17 |
| CBR at 95% of MDD, % | 43 |

Figures Captions

Fig.1 : X-ray diffraction patterns of the lime mixes samples

a- Raw ; b-2wt.% lime ; c-3wt.% lime ; d-8wt.% lime
K : kaolinite ; Q : quartz ; G : goethite ; H : hematite ; R : rankinite ; C : calcite ;
CH : portlandite.

Fig.2 : DTA curves of the lime mixes samples

a- Raw ; b-2wt.% lime ; c-3wt.% lime ; d-8wt.% lime
CSH : calcium silicate hydrate ; O : organic matter ; C : calcite ; CH : portlandite ; G : goethite.

Fig.3: Infrared spectra of the lime mixes samples

a- Raw ; b-3wt.% lime ; c-8wt.% lime

Fig.4 : Relative intensity of the infrared bands located at 3690 and 3620cm⁻¹ versus lime contents

Fig.5 : SEM images and EDS analyses of the quoted zones

(a , b)- Raw; c-2wt.% lime; d-3wt.% lime; e-8wt.% lime

Fig.6 : Grain size distribution of the lime mixes samples

Fig.7 : Evolution of PI and MBV with lime additions

Fig.8 : Evolution of pH versus lime contents

Fig. 9 : Evolution of OMC and MDD with lime amounts

Fig. 10 : CBR evolution with lime increasing

A : CBR value for samples immersed for 4 days; B : CBR value of dried in air for 3 days and immersed for 4 days samples

Fig. 11 : Variations of compressive strengths with lime contents and curing times

Fig.12 : Variations of tensile strengths with lime contents and curing times

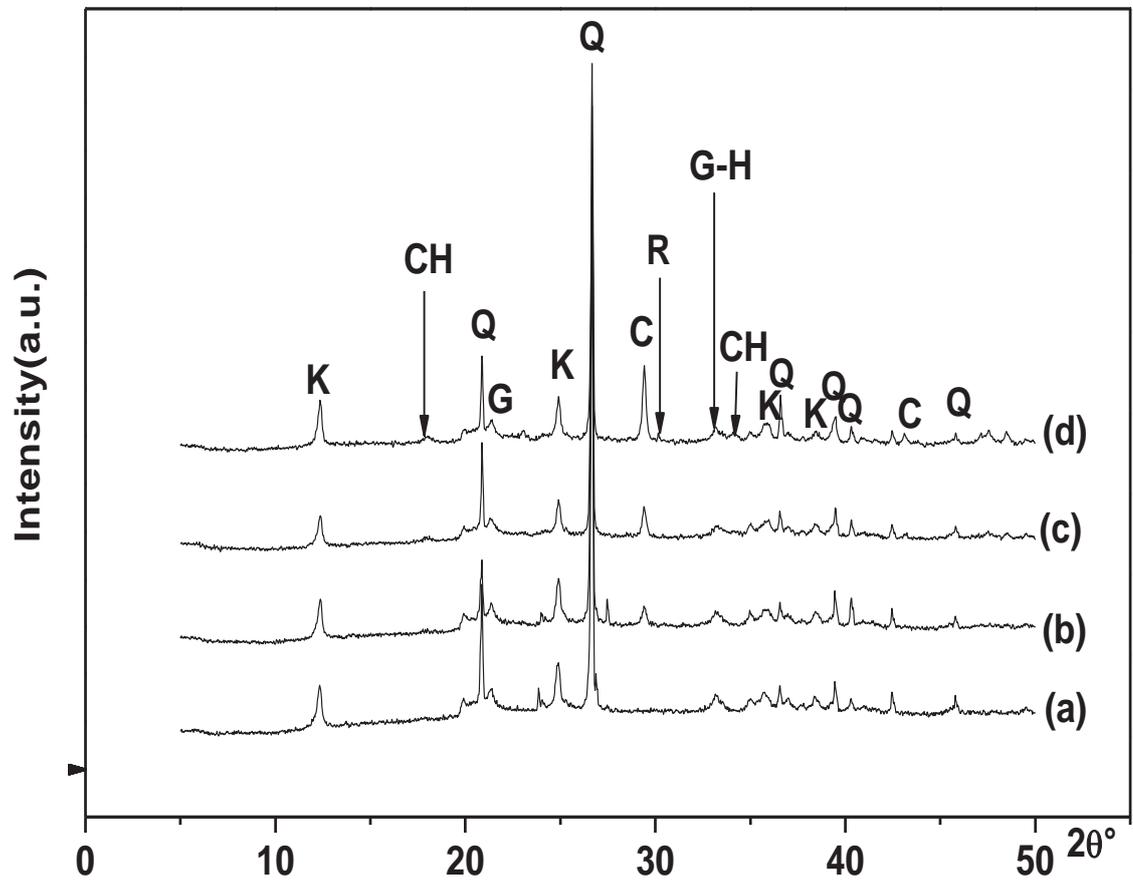


Fig. 1

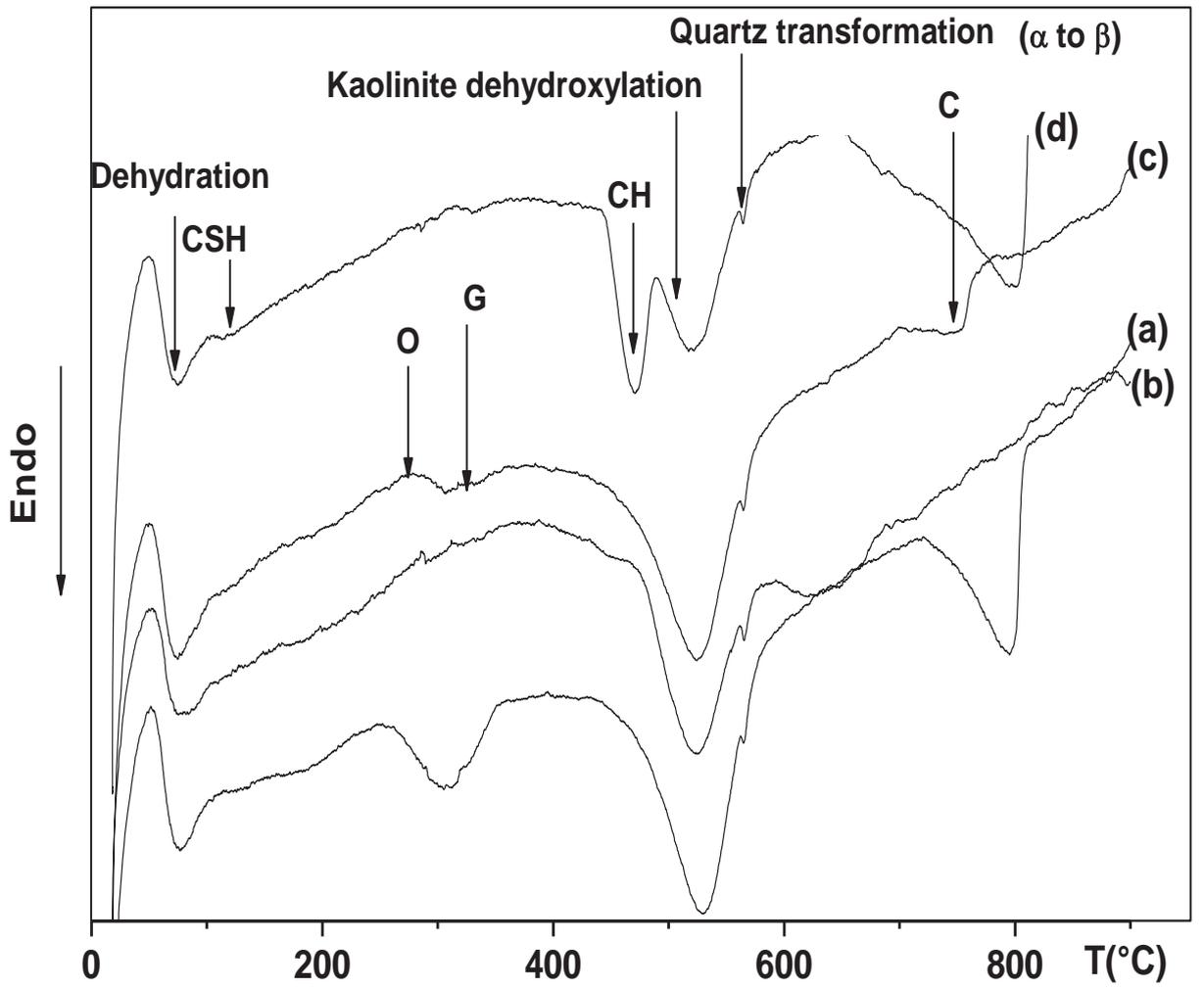
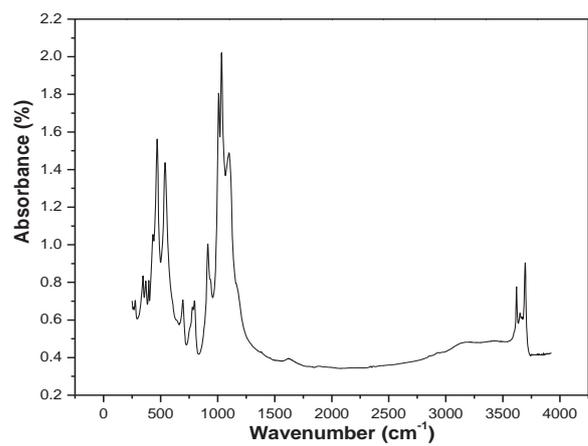
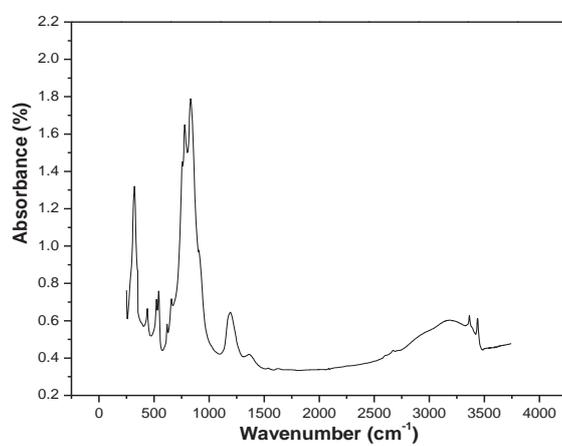


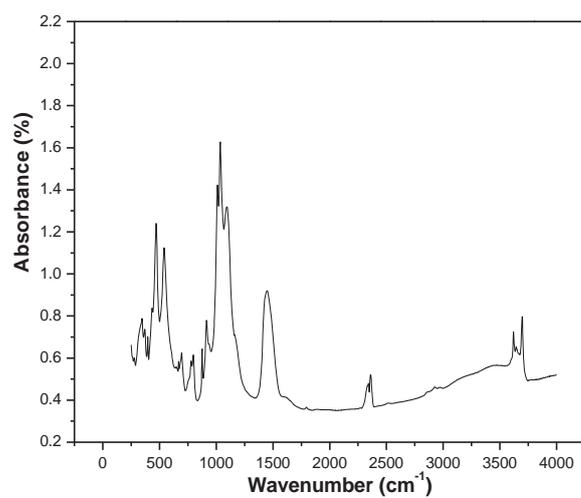
Fig.2



a



b



c

Fig.3

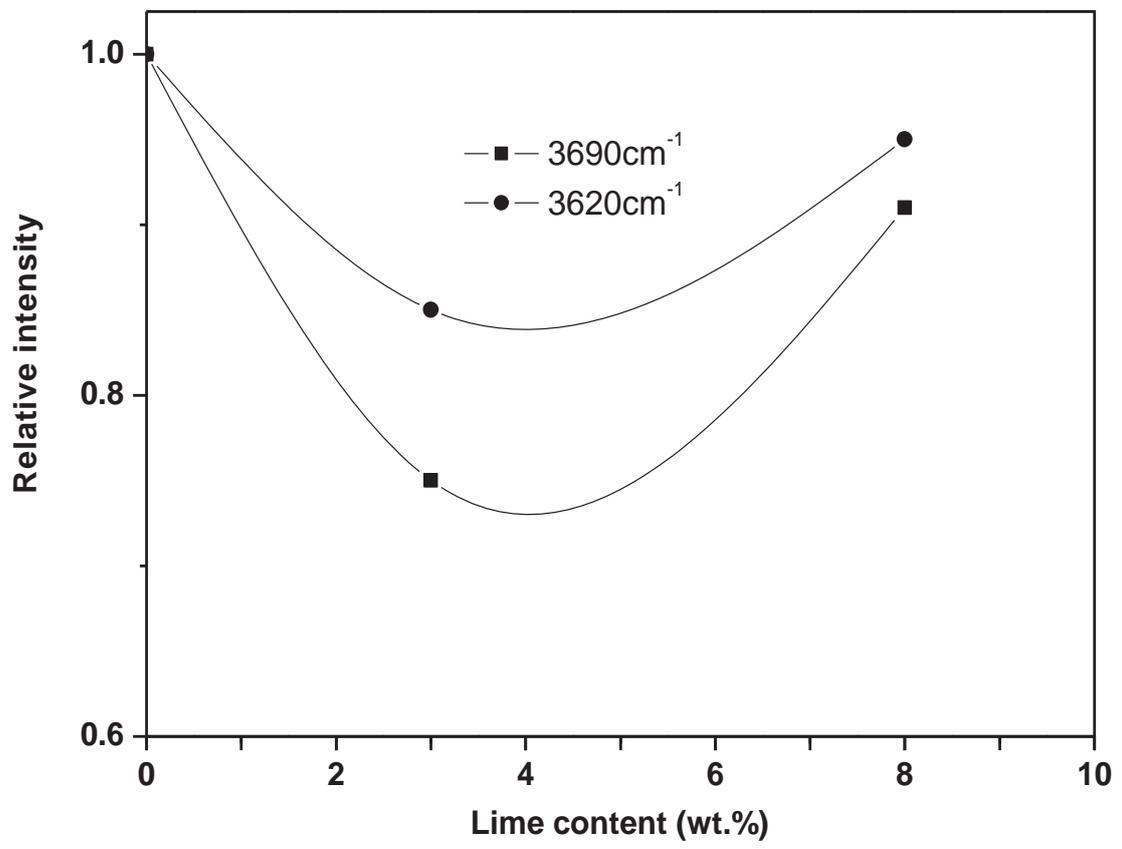


Fig.4

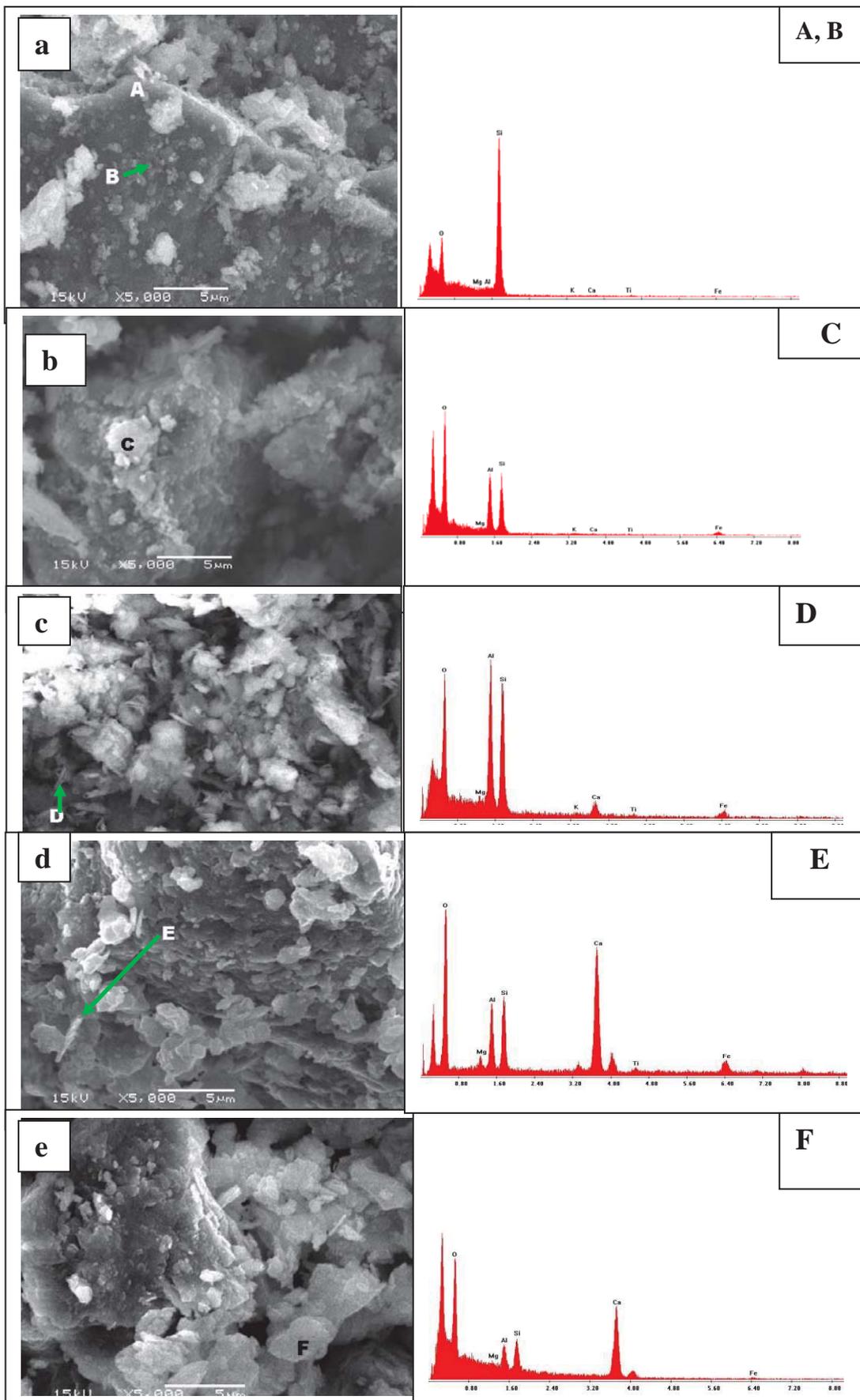


Fig.5

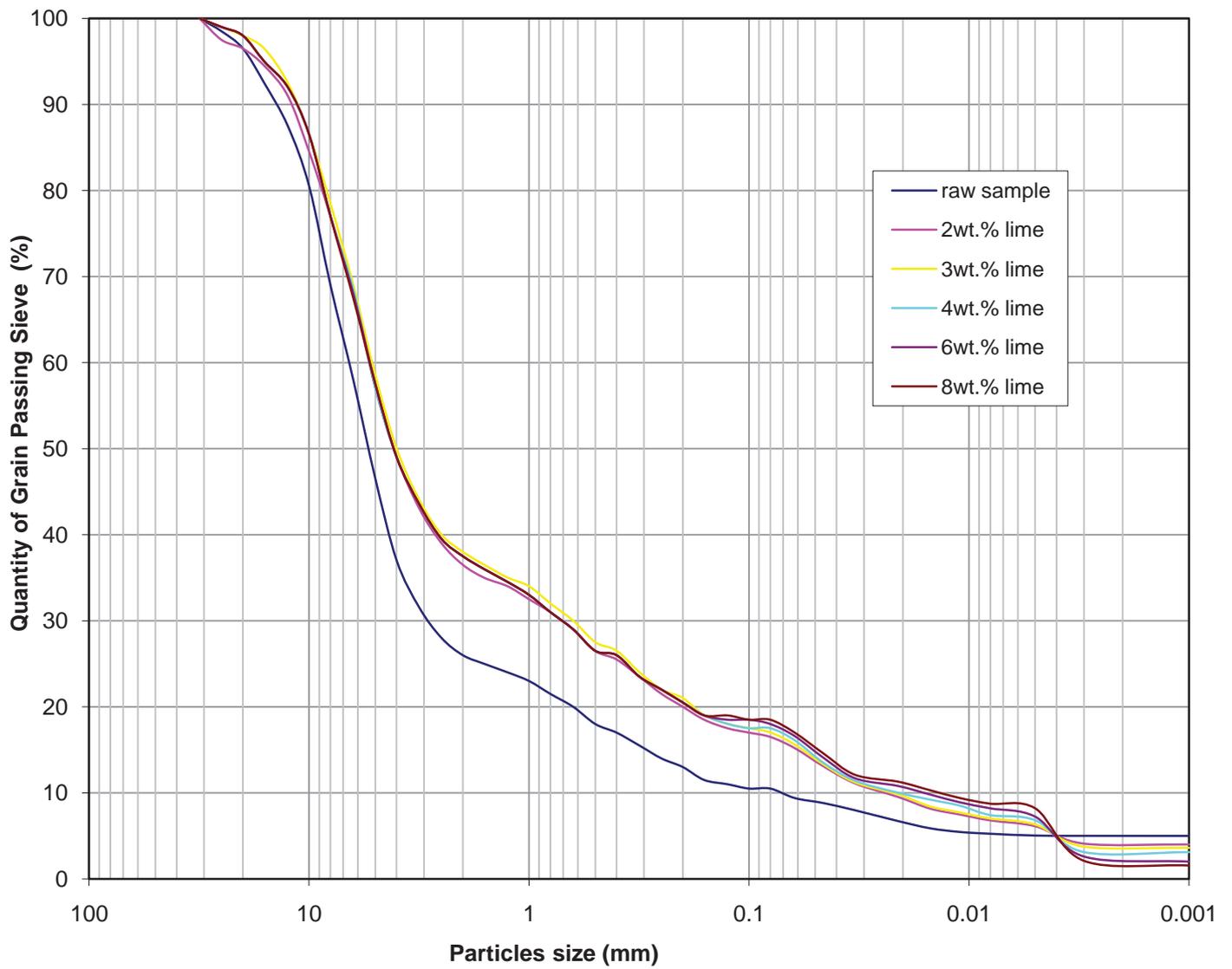


Fig.6

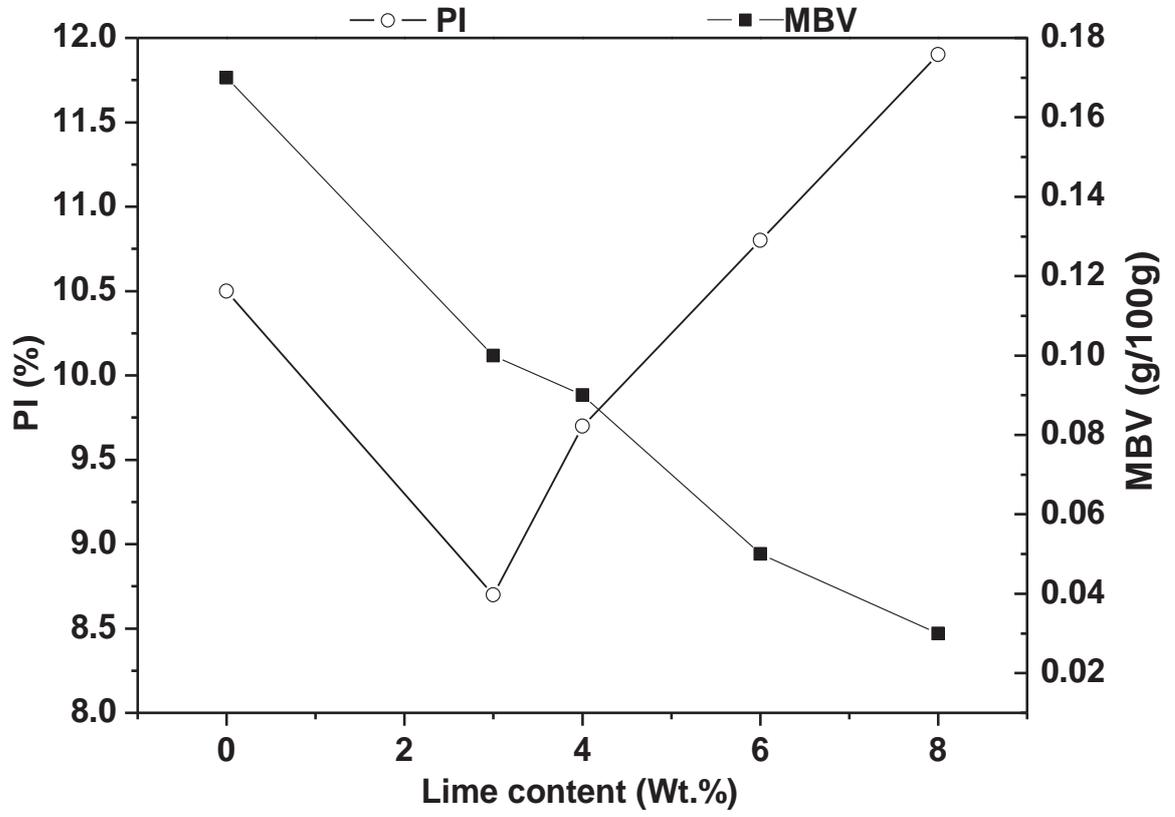


Fig.7

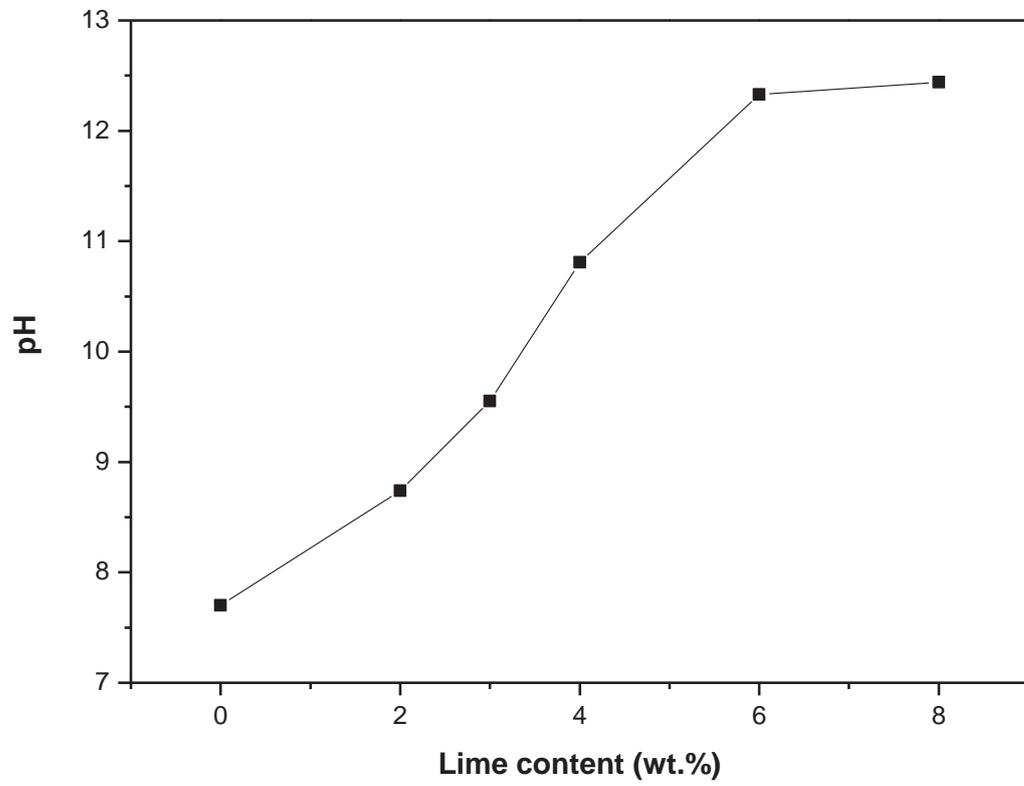


Fig.8

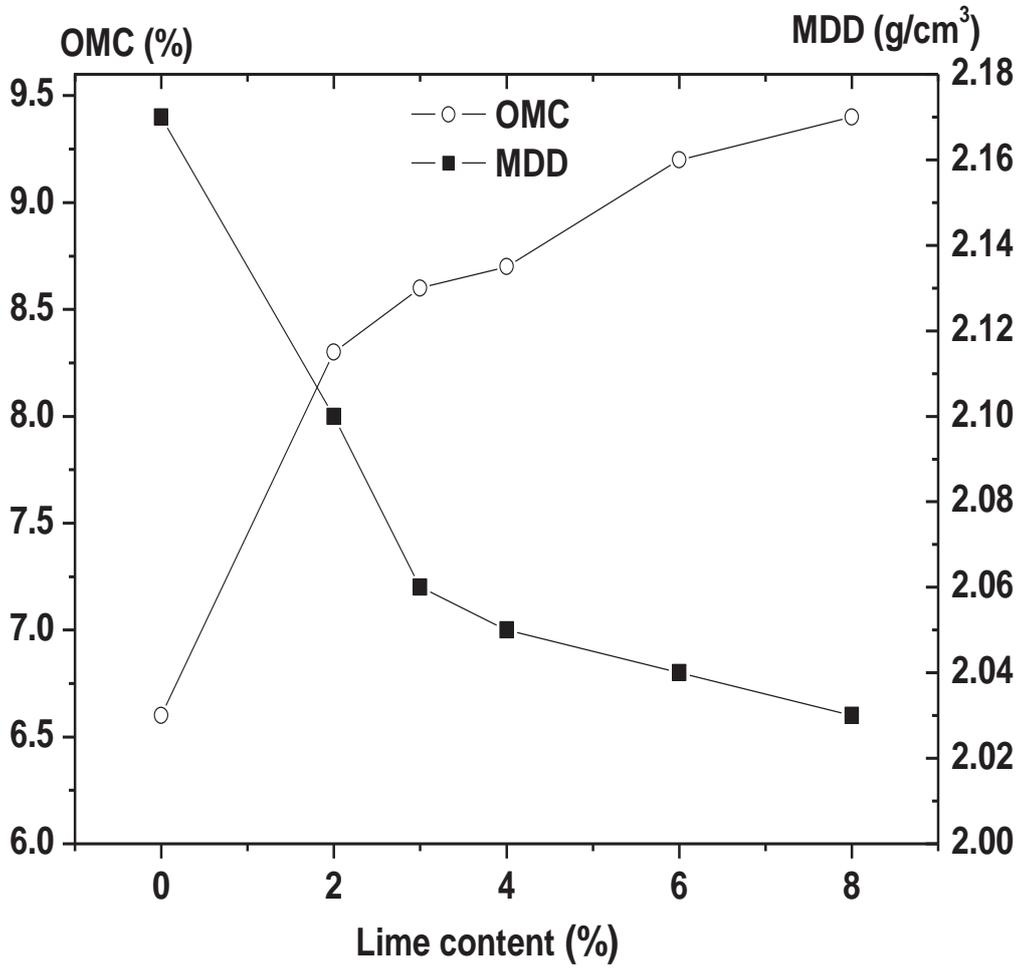


Fig.9

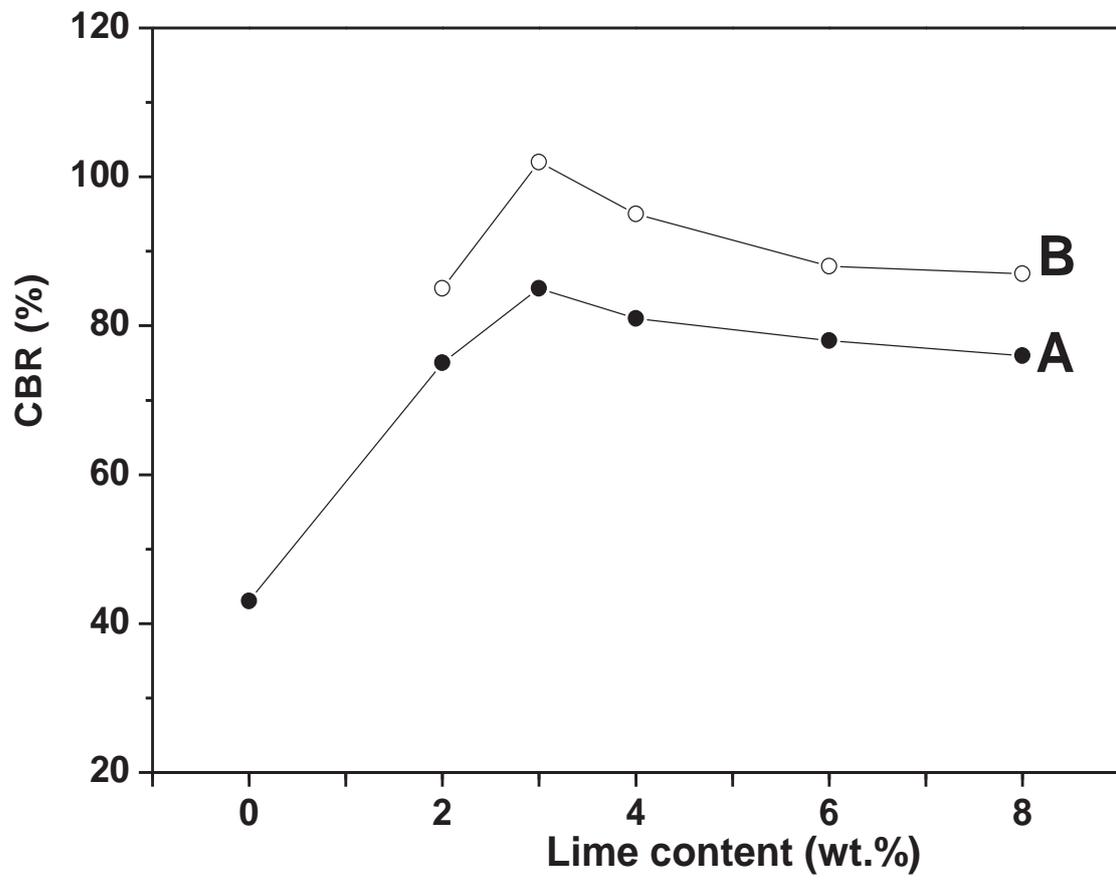


Fig.10

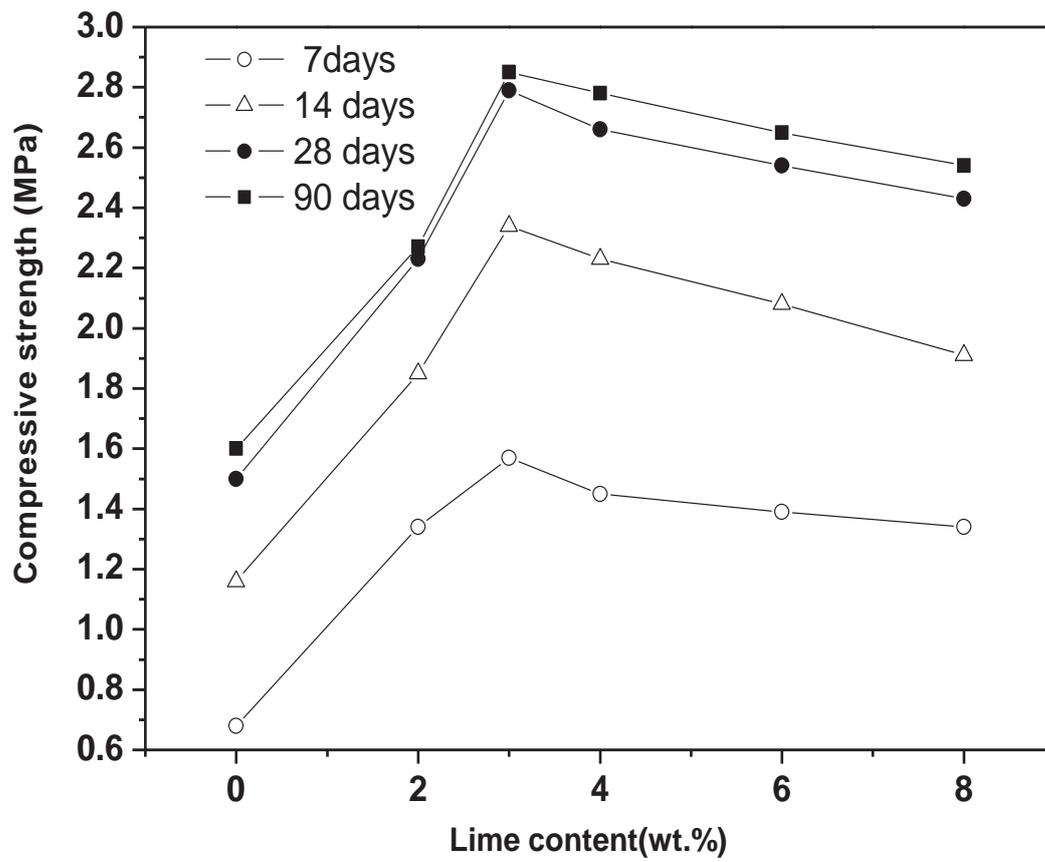


Fig.11

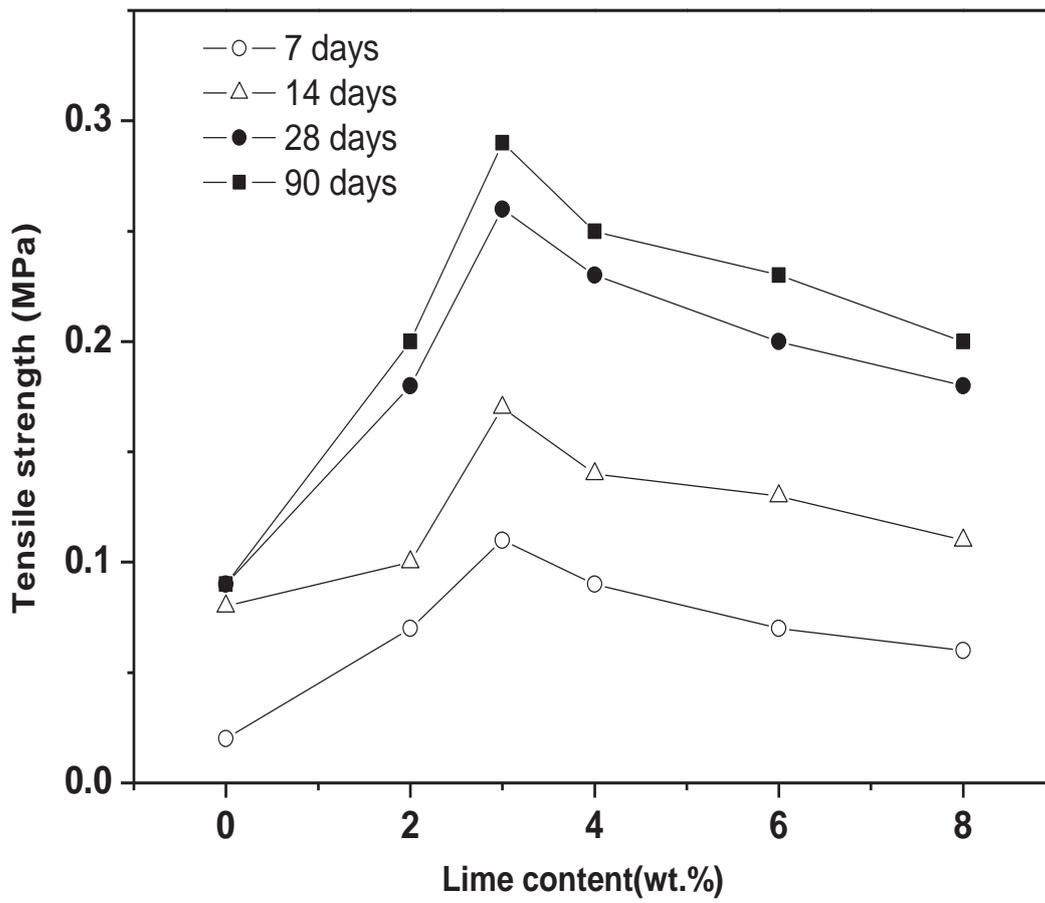


Fig.12