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African Journal of Environmental Science and Technology

Full Length Research Paper

Identification and classification of clayshale characteristic and some considerations for slope stability

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It is very necessary for clay shale layer to slake for durability. The identification and classification of clayshale should be considered based on slope design. Several locations of clayshale slopes were stable during the construction of Cipularang Highway 97 + 000 km, West Java, Indonesia. However, they experience landslides after some time. Therefore, material properties and shear strength characteristics of clayshale layer need to be reevaluated. Laboratory test result indicated that all sample can be classified as overconsolidated –CH or indicated as A–7–6 based on the AASHTO standard. Result of triaxial test also produced significant difference of shear strength parameters of *(c)* dan *(□)* from bore holes. Thus, slake-durability and rate of slaking tests need to be performed. The clayshale sample can be categorized as *“*slightly weathered” class and indicated as “slow rate” of weathering process.

Key words: Clayshale, slake-durability, rate of slaking.

INTRODUCTION

Toll highway of Cikampek-Padalarang or Cipularang (Figure 1) was designed to anticipate traffic increase in cities such as Jakarta and Bandung. This toll highway that connects Jakarta-Cikampek and Padalarang-Cileunyi (Padaleunyi) toll highways has been operated earlier (Jasa Marga (Persero), Tbk, 2003).

Topography of Cipularang area is not flat and there are many steep cliffs (Figure 2). Steep cliffs are located at the western and eastern parts from the main body of Toll Highway which has a low soil shear strength at the slopes. Some landslides occur frequently at 97 + 000 km from Jakarta.

From some field observations, landslides are caused by some movements of “clayshale” layers. Collapsed block model from soil surface is always found in the study area and soil mass is usually accumulated at the toe of slopes. Actually, during construction, these clayshale layers were seen as unsuitable materials or not used and always cut and filled at the toe of embankment as a counter weight or in a disposal area.

Clayshale is an *argillaceous* material, an over-consolidated or a compacted soil, rock and dust that

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Figure 1. Toll Highway plan in Java Island, Indonesia (Jasa Marga (Persero), Tbk, 2003).

undergoes various types of sedimentation, consolidation, and cementation process. Clayshale is formed by clay minerals and claystone (Terzaghi, 1967; Franklin, 1981; Bates and Jackson, 1983). The term *"shale"* has been applied to a class of materials that are generally described as fine-grained, and/or the commonest type of sedimentary rock. Shale is one of the common transition materials in all layers of 50 to 75% of the earth's surface (Leet, 1971).

Clayshale is well known as a degradable material, easily fragile, and has low level of durability (Taylor, 1948). Clayshale itself is sometimes considered a rock but, when it is exposed to air or has the chance to take on water, it may rapidly decompose (Piteau and

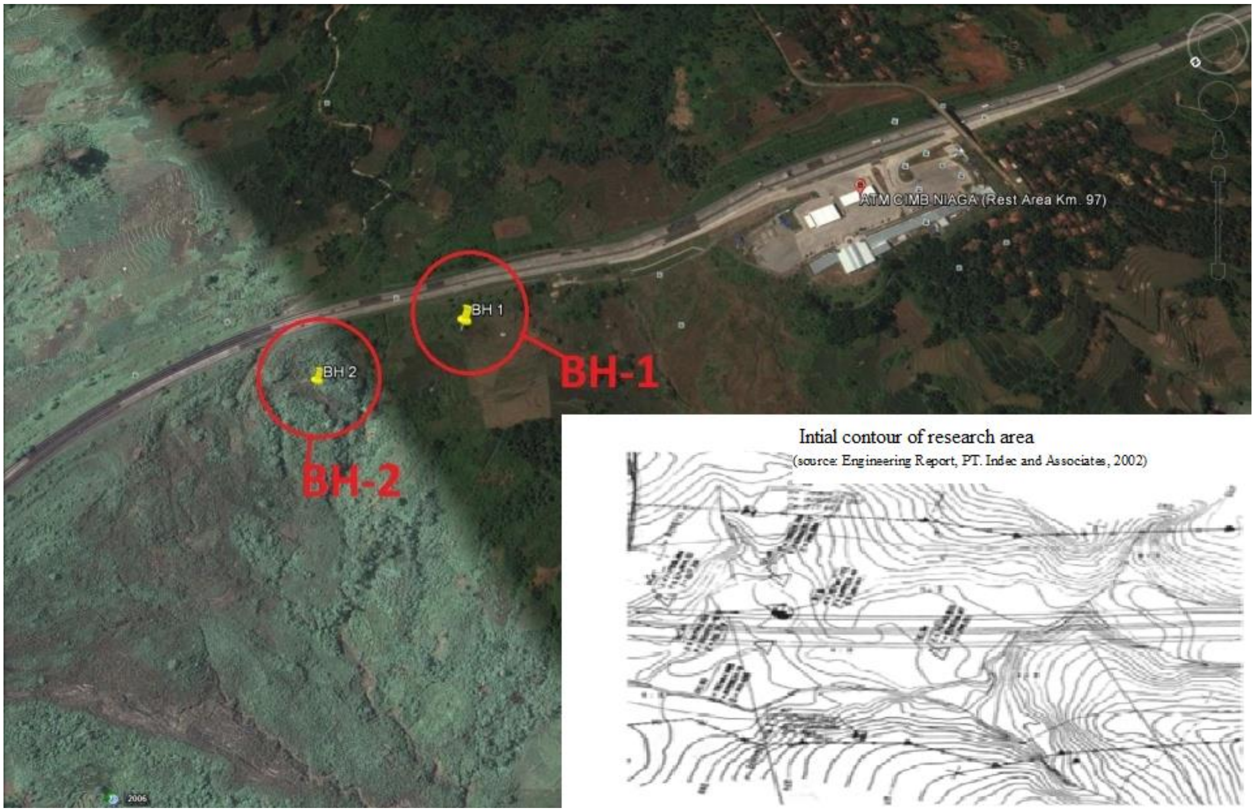

Figure 2. Cipularang research area.

Peckover, 1978). Clayshale always brings out some issues in geotechnical engineering, such as the selection of embankment material, bearing capacity of shallow and deep foundations, natural and artificial slope stability, piping and ground settlement.

Study area

Geological history began with deposition of claystone and limestone by a deposition system and compression process at the end of Miocene from the depth of 0 to 200 m. In the Plio-Pleistocene era occured the tectonic activity and produced the anticline folds form on the claystone structure (LAPI-ITB, 2006).

Faults can be identified through analysis of remote-sensing and also field geology study (Figure 3). In research area, there are two types of fault, that is thrust fault and strike-slip fault. Crossing of two kind of thrust fault and strike-slip fault is caused by a weakness of those areas. Fold structures occured at Jatiluhur formation, forming the syncline and anticline with the fold axis in the east-west direction. There were also many fault structures within the north-south and southwest-southeast directions with normal and shear faults, formed early by syncline and anticline in those directions. More kinds of landslide are developed at cross of two faults. Indication of strike-slip fault at clayshale is shown at the upper hill. At the foot hill, cracks and joints are shown by curve of strike-slip fault. Kinds of landslide are slump, topple and fall.

Generally, geology structure in the study area in Cipularang Toll Highway consists of alluvium-dilluvium deposit, tuffacious siltstone, sandstone, conglomerates, and tertiery clay formation. Mostly, gravitational collapse occured on this formation. In 1973, one ground movement occured at Ciganea, Purwakarta (Soewartojo et al., 1973), where the heavy landslide material moved and pressed claystone, and finally resulted in heaving on claystone layers at the bedrock. Details on the geology of the Cipularang shale are presented in Figure 3. From Figure 3, these layers are nearly flat dip and strike to the opposite layers, which indicates that the layers are marine sediment. These layers consist of alternating siltstone, sandstone and conglomerates with the maximum thickness of 1.0 m. In dry condition, these layers would be dense, however, most silt and sand layers on that formation tend to be loose in saturated condition. Tertiery clay formation is more commonly recognized as Subang formation. This formation consists

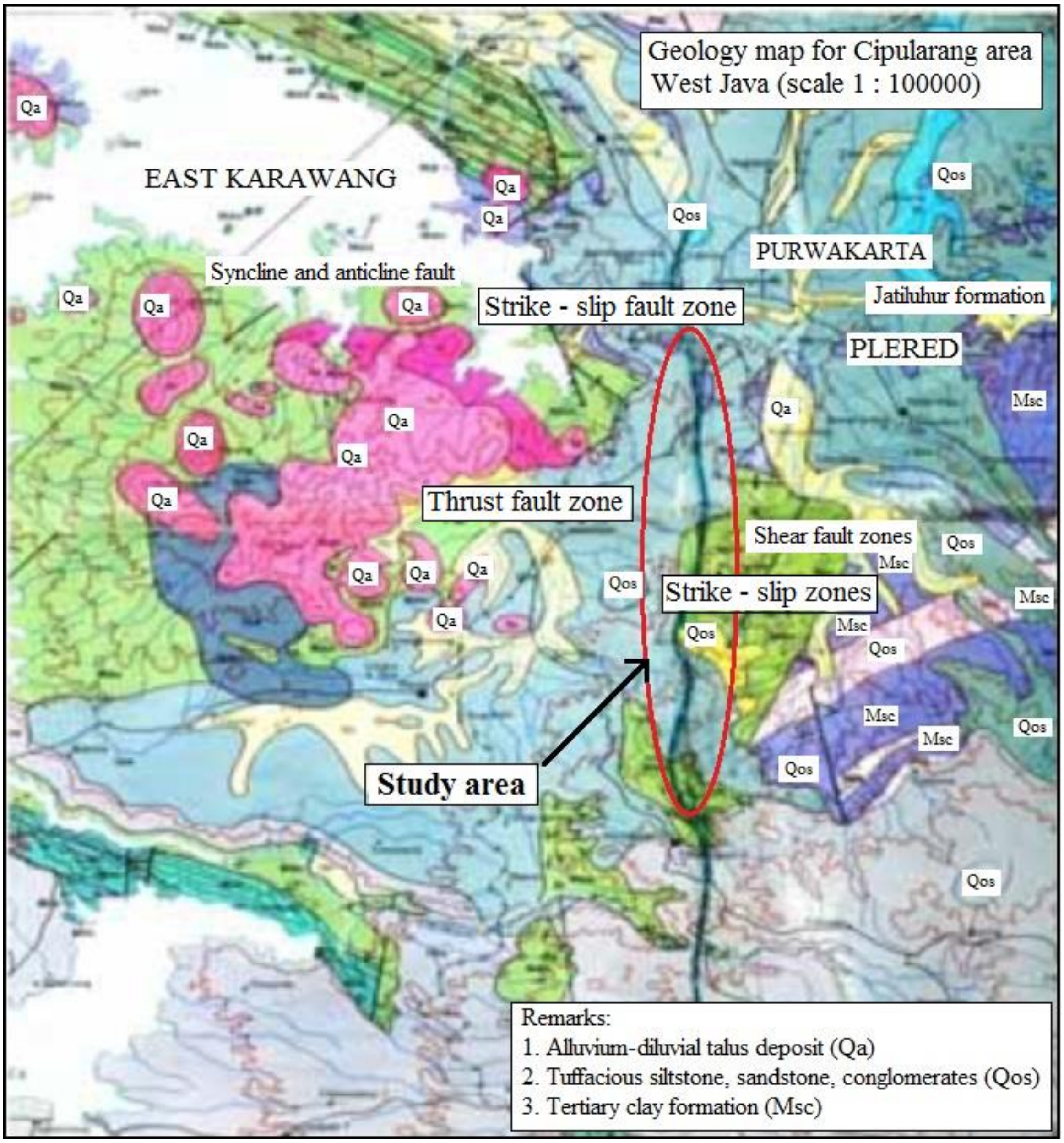

Figure 3. Geological map of the research area (Direktorat Geologi Departemen Pertambangan, 2002).

of a uniform claystone with the dark grey color. Characteristic of clayshale layers is hard, although there is low shear strenght of thin layer.

Basic theoretical

Clayshale as degradable materials

Clayshale is also one of the most complex materials from the point of view of geology, geotechnical, and environmental science. Clayshale exhibits special behavior having a tendency to change very rapidly from rock into soil in a relatively short time. Thus, clayshale, most often, is regarded as problem materials. In most cases, the formation of clayshale is influenced by the type of constituent mineral, as well as the climate condition, physiography and topography of the area under consideration (Deen, 1981).

Most clayshale exists on unsaturated zone since capillary effects play more role on that area and depend on location of ground water. In addition, clayshale is

Figure 4. Clayshale in Toll Cipularang Highway (Jasa Marga (Persero), Tbk. 2003).

included as an intermediate or transition material between soil and rock. Clayshale is the compaction of clay, silt, sand and also dust with various types of cementation.

Clayshale is a layer of fine-grained sedimentary rocks formed from consolidated soil as a result of great pressure or tension in the past. Sedimentary rocks (clayshales in particular) are typically formed relatively near the earth's surface and without extreme heat and pressure that occur at depth. They tend to be minerallogically stable near the surface. They involve the weathering of these materials and either a reversal of the consolidation pressure or dissolution of cement bonds holding the grains or mineral groups together (Walkinshaw and Shanti, 1996).

Technically, the main behavior is very hard to analyze, but when they are exposed to sunlight, air and water, then in a relatively short time they become very soft. One example of clay shale terrain in Indonesia is Cipularang Highway (Figure 4).

Cipularang clayshale exists on unsaturated zone; the capillary gives strength to clayshale where the ground table water is not found until the boring works during soil investigation. Capillary forces emerged. This is caused by negative pore water pressure and can crush clayshale mass if the clayhale bonding is inadequate. Finally, it can behave as soil.

Geologically and geotechnically, clayshale classification (Wenworth, 1922; Ingram, 1953; Underwood, 1967; Folk, 1968; Terzaghi, 1936; Bjerrum, 1967; BSI, 1957; Gamble, 1971; Deo, 1972; Morgenstern and Eigenbrod, 1974; Botts, 1998) has been proposed and shown that clayshale exists in a classification zone unclear and complex. Thus, most clayshale could be classified as transition material between soil and rock.

Identification of clayshale behavior

Clayshale material has a range of strength potential between soft soil and low quality of rock. Keller (1976) stated that an unixial compression (q_u) of clayshale could reach less than 1.80 kg/cm^2. Peck et al. (1974) found that a range of the uniaxial compression of fresh clayshale exposed by air is 280 to 2250 kg/cm^2. Attewell and Farmer (1976) concluded that clayshale had a low sensitivity. Besides that, clayshale strength depended on level and kind of bonding material.

US Army (1956), Bjerrum (1967), and Johnson (1969) included that clayshale behavior is an overconsolidated clay. They also stated that the geology history begins from time deposition to recent condition as unweathered or weathered clayshale. Both those clayshales had different characteristic. Weathering could be defined by all changes occurring near surface of soil/rocks.

Typical elasticity modulus of clayshale (US Army, 1990) behaving like soil material ranges between 100 and 200 kg/cm^2. Determination of modulus could be taken by pressuremeter, uniaxial, or ultrasonic testings. Modulus value is also influenced by anisotropic characteristics of soil/rock. On clayshale case, comparison of E_v/E_h could be less than 1.0. Vargas (1953) reported that modulus value between vertical and horizontal directions for hardest clayshale is 0.65.

Hendron et al. (1968) conducted some research to compare elasticity modulus obtained by uniaxial and pressuremeter testings. They concluded that elasticity modulus obtained laboratory works using uniaxial and pressuremeter testings are three times lower than field investigation. Besides that, change of water content is very sensitive to modulus values. Therefore, increased water content could decrease modulus values.

Diagenetic process

Soil is a compressible material based on clay mineral content. In consolidation soil testing, there is a virgin section (AB) during loading in progress and a rebound section (BC) during unloading in progress (Figure 5). The

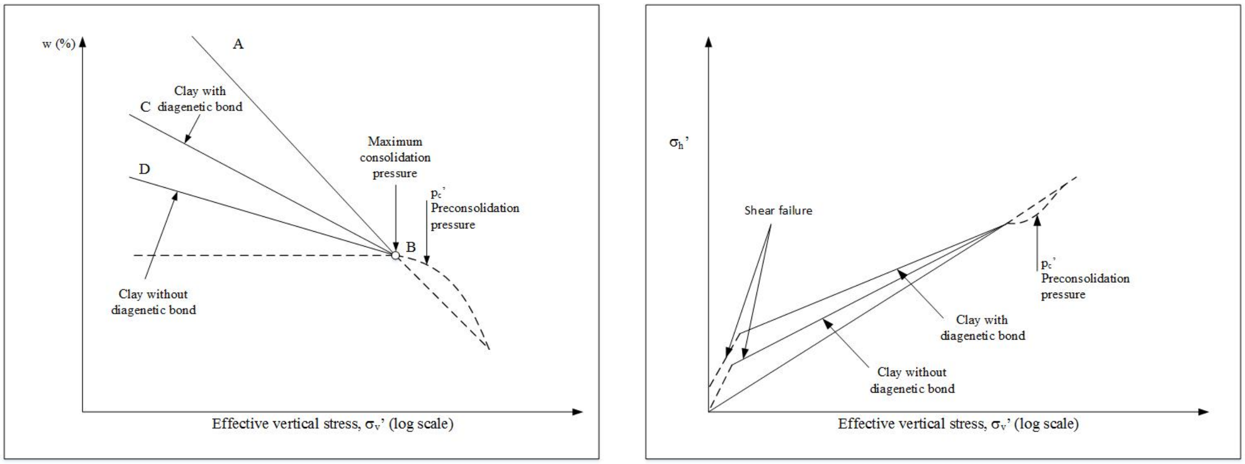

Figure 5. Geological history for overconsolidated soil (OC) (Bjerrum, 1967).

section of rebound shows a recoverable characteristic of soil influenced by clay particle form. When clay is consolidated on specific stress, it would have a recoverable strain energy. Total strain energy would be influenced by consolidation stress and degree of clay plasticity. The higher the degree of clay plasticity, the higher the total strain energy.

From the instant clay sediments are deposited, they are subjected to physical (overburden pressure, time, and temperature, compaction, etc., and chemical (fluid, organic matter, etc.) forces. The combined effect of physical and chemical alterations during time of sedimentation to regain equilibrium is called *"diagenesis."* Diagenesis comprises all process that convert raw sediment to rock (especially for sedimentary rock). These reactions can enhance, modify or destroy porosity and permeability. Existence of diagenetic can result in interparticle contact area that has a large stress and a chemical recrystallization process of a solid to form large crystal grains from smaller ones. When chemical composition changes, adhesion would emerge on interparticle contact area. Furthermore, interparticle bonding on contact area can be created by cementing agents.

Therefore, in natural condition, clay under large pressure and a long time period without volume change becomes stronger and more brittle. Strength of diagenetic bonds depends on consolidation stress and mineral composition, pore fluid, time, and temperature, and also variation in strength. The strong bonds generate clay in *hardening* behavior and classified as soft rock.

Diagenetic bonding effect could increase durability of clay to volume changes caused by enhancement load. Figure 5 shows that an increament load value before t p_c could not result in a volume change significantly. p_c value is determined by durability combination of volume changes without bonding agents and residual stress resulting from diagenetic bond of clay. However, p_c value obtained from laboratory consolidation test is limited only to clay unaffected by genetic features.

With time, clay desposit undergoes an unloading time caused by erosion that reduces overburden pressure. As a result, strain energy inducing clay deposit will have a tendency to swell and increase water content (w). However, that swelling is limited by interparticle bonds. The high unloading level due to increased stress generates the dispersed interparticle bonds. Therefore, the water content will increase near the ground surface. Final water content equilibrium depends on strain energy, bonds strength, and damaged interparticle bonds. Rebound curve of BD shows the equilibrium of water content during unloading process on clay with the strong bonds, while BC curve applies to clay without bonds.

Weathering process

Weathering process on overconsolidated clay with diagenetic bonding can be distinguished in 2 (two) phases, such as:

(a) Disintegration phase: The phase where clay structure is scattered due to vanished interparticle bond caused by strain energy. Disintegration of soil structure is due to soil swelling potential.
(b) Chemical change phase: The phase where decomposition of clay mineral occurs.

From Figure 6, in clay with strong bonds, disintegration phase occurs more rapidly and physical properties become important. The main effect of disintegration is the gradual damage of particle bonding. When these

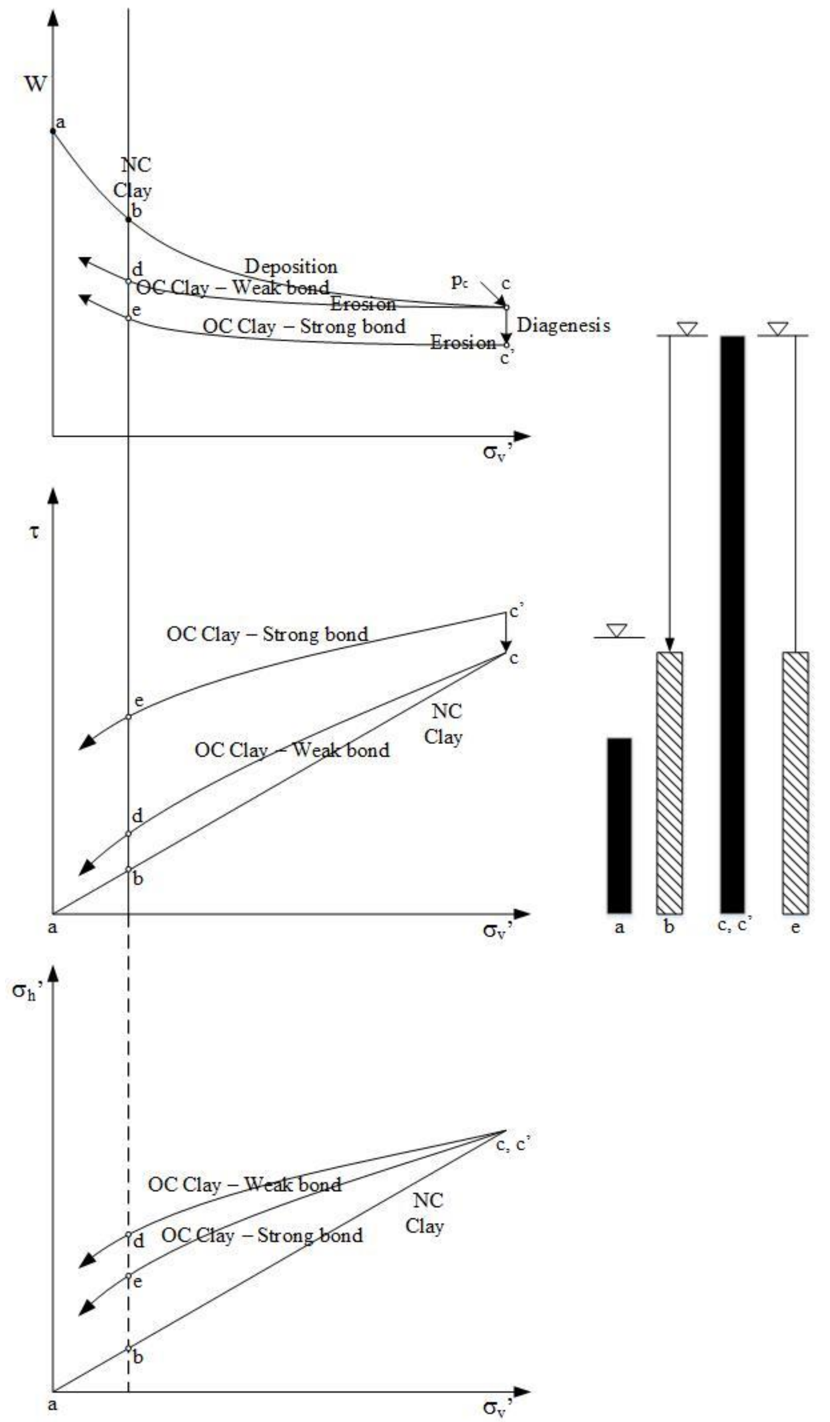

Figure 6. Geological history for normally consolidated (NC) and overconsolidated (OC) clay (Bjerrum, 1967).

bondings are damaged, stored strain energy leads to the swelling potential of clay, increased water content, and low strength. Due to horizontal structure, soil swelling has an upward direction. Consequently, vertical effective stress increases until the strain energy is exhausted.

Occurrence of swelling depends on the stored strain energy. If diagenetic bonds is very weak, the strain energy is dissipated during unloading process and produces the largest swelling potential during disintegration process.

The main cause of decayed diagenetic bonds is strain that occurs due to physical changes. Strain becomes smaller with the depth increasing, so that it can be divided by zones depending on disintegration level. Bjerrum (1967) classified these zones into 3 (three) parts, such as: zone of complete disintegration; zone of advanced disintegration; and zone of medium disintegration.

Top zone and near the ground surface is zone of complete disintegration influenced by alternating temperature change, drying and saturation process. This zone is affected by chemical process, like oxidation process and mineral decomposition. The water content and soil strength of this zone is greatly dependent on climatic condition.

Below the zone of complete disintegration is followed by zone of advanced disintegration. This zone is greatly influenced by variation of effective stress as a result of pore water pressure change caused by ground water table fluctuation. It depends on surface of topography; shear strain plays an important role in the disintegration process.

Zone of advanced disintegration has an open crack system. Capiller stress from water circulation on this crack induces the slaking occurence in clay formation. On this zone, there is always a chemical change. In general, soil formation of this zone is softer and has higher water content compared to zone below, zone of complete and advanced disintegration.

At the bottom, there is zone of medium disintegration. Strain is larger on definite depth of location beneath slope than relatively flat surface. Study performed by Einsele and Wallrauch (1964) and Attewell and Farmer (1976) show the water content varies greatly for this zone. The variation reflects mineral composition on clay indicated by relating with liquid limit (LL).

Number of strain energy depends on type clay mineral where it can generate interference volume in the surrounding clay and disintegrated diagenetic bond. Strain and energy dissipation is not equal and rises to cracks and fissures.

Clayshale classification and general nature

Classification of clayshale has been discussed geotechnically and geologically. In general, argillaceous rocks such as shale, mudstone, claystone, siltstone, and clay shale are characterized by wide variations both in their engineering properties and composition. The common characteristics of this group of rocks are that all members are fine-grained and composed predominantly of clay and silt sized materials. The term shale has been used by some authors for all argillaceous rocks, including claystone, siltstone and mudstone (Ingram, 1953; Krumbein and Sloss, 1963). Others have specified the large group as the mudstone group and classified shale as a member of this group (Twenhofel, 1939; Muller, 1964). Terzaghi (1946) had a different opinion in defining shale. He claimed that the material should be called shale when it displayed a clear ring upon striking by a hammer and showed no change in volume when it was immersed in water. Many classifications used for argillaceous rocks are geological and depend on such properties as quartz content, grain size, colour, and the degree of compaction. Although, these provide important information regarding the geological history of these materials, such classifications can be misleading when concerned with engineering behavior. This is particularly evident when evaluating the behavior of clay shales.

The general characteristics of clay shales include: (1) highly overconsolidated, (2) commonly small scale fissured, (3) strong diagenetic bonding, (4) tendency to slake when rewetted after drying, (5) high swelling pressure in the presence of water, and (6) significant disintegration as a result of interaction with water.

Beyond this general description of clay shales, the classification of these materials has become complicated and confusing. Numerous classification schemes for argillaceous materials have been proposed, and have been reviewed by Shamburger et al. (1975), Deen (1981), and others. Classification of clayshale actually is fairly complex and as an intermediate material between rock and soil (Botts, 1986).

Geological classification

The major objective of geological classifications is the determination of the geological history of deposits. Initially classification (Wentworth, 1922) was based primarily on grain size and arbitrarily set the boundary between argillaceous material and the remaining sedimentary rocks. Ingram (1953) took the classification one step further; he subdivided all clayey materials based on percentages of silt and clay components, and on their breaking characteristics. Ingram used the term fissility which is the fine scale fracturing in the shale surface to distinguish shale from stone, while the prefixes “clay”, “silt”, or “mud” are derived from the relative percentages of the grain size components. Thereafter, such terms as claystone, siltstone, and clay shale began to be used in the literature.

In an attempt to distinguish between compacted and cemented shale, Philbrick (1950) performed a simple weathering test that was based on five cycles of drying and wetting. He suggested that the shales that reduced to grain sized particles be termed compacted shales and those that were unaffected be termed cemented shale. This approach followed earlier classification by Mead

Table 1. British Standard Institute classification (1957).

Consistency	Field indication	Strength (q_u) (kN/m^2)
Very stiff	Brittle or very tough	> 150
Stiff	Cannot be molded in fingers	75 - 150
Firm	Molded in fingers by firm pressure	40 - 75
Soft	Easily molded in fingers	20 - 40
Very soft	Extrudes between fingers	< 20 kN/m^2

(1936) who classified shales according to their cementation into two broad groups, the first is compacted shales that have been consolidated under stress by the overlying sediment without intergranular cement, and the second is cemented shales that could have a cementing agent (calcareous, siliceous, or ferruginous) or a bonding material formed by recrystallisation of clay minerals.

A similar division by Underwood (1967) introduced new terms, “soil-like” shale for compacted shale and “rock-like” shale or bonded shale for cemented shale. Although the classification was aimed to serve geological purposes, the division between these two groups is poorly defined. This shortcoming motivated Folk (1968) to clarify Ingram’s scheme by refining “mudstone” as argillaceous materials with sub-equalamounts of clay and silt. This was further modified by Gamble (1971) who introduced a classification scheme that was essentially the same as Ingram’s except that the terms clay shale and silt shale have been changed into “clayey shale” and “silty shale”. Although, this change may seem insignificant, the term clayey shale does help to distinguish a clay rich shale from a clay shale which, in engineering usage, implies certain engineering behavior and not simply a fissile rock which is rich in clay content. Based on stress history, Bjerrum (1967) classified shales as overconsolidated plastic clays with strongly developed diagenetic bonds and clay-shales as overconsolidated plastic clay with poorly developed diagenetic bonds. Similarly, Skempton and Hutchinson (1969) attempted to crudely relate geological origin of materials to their potential engineering behavior. However, the usefulness of their scheme for purposes other than for providing a general understanding of possible relationships is quite limited. Although, these geological classification schemes can provide some useful information for engineers, they are generally inadequate for evaluating potential engineering behavior of clay shale. Nevertheless, the above review indicates the use of the term “clay shale” in the geological sense to generally describe a fissile rock, rich in clay-sized components. However, the use of the term clay shale does not carry the same meaning when it is used in the engineering literature.

Based on some authors and some engineering literatures, there are obtained for this study that geological classification is based on gradation of grain size, clay fraction, and crushed rock characteristics as suggested by Wenworth (1922), Ingram (1953), Folk (1968), and Underwood (1967). This information is useful for determination of clayshale description, even though classification of clayshale actually is fairly complex and as an intermediate material between rock and soil. However, from geological classification in general it can be found that clayshale has an *‘transition properties’* between rock and soil.

Geotechnical classification

The basic purpose of an engineering classification is to provide terms that aid the user in distinguishing materials which have similar engineering properties. The more recent classification schemes for argillaceous materials have attempted to account for their potential engineering behavior. However, classification of argillaceous material for engineering purposes has been particularly difficult. The difficulties arise from the transitional nature of some of these materials. This transitional nature creates confusion among many geotechnical engineers who are accustomed to viewing a material as either a rock or a soil, but not as a material that can have properties of both. An early engineering classification was proposed by Terzaghi (1936) that divided clays based on stiffness and the presence or absence of fissures into three major terms; soft clays free from fissures, stiff clay free from fissures, and stiff fissured clay. Bjerrum (1967) adopted a different approach, he proposed an overlapping three-fold classification, based on bond strength and extending up to shale materials. In his classification, these descriptive terms were followed: (a) overconsolidated clays with weak or no bonds, (b) clay shales, that is, overconsolidated clays with developed diagenetic bonds, and (c) shale, that is, overconsolidated clays with strongly defined diagenetic bonds. The two classifications have significant, but poorly distinguished overlap between them creating some confusion of terms. Further confusion has developed from the use of the British Standard Institute classification, which uses similar terms based on consistency or strength (Table 1, British Standard Institute classification). These classifications caused some ambiguities particularly when using terms

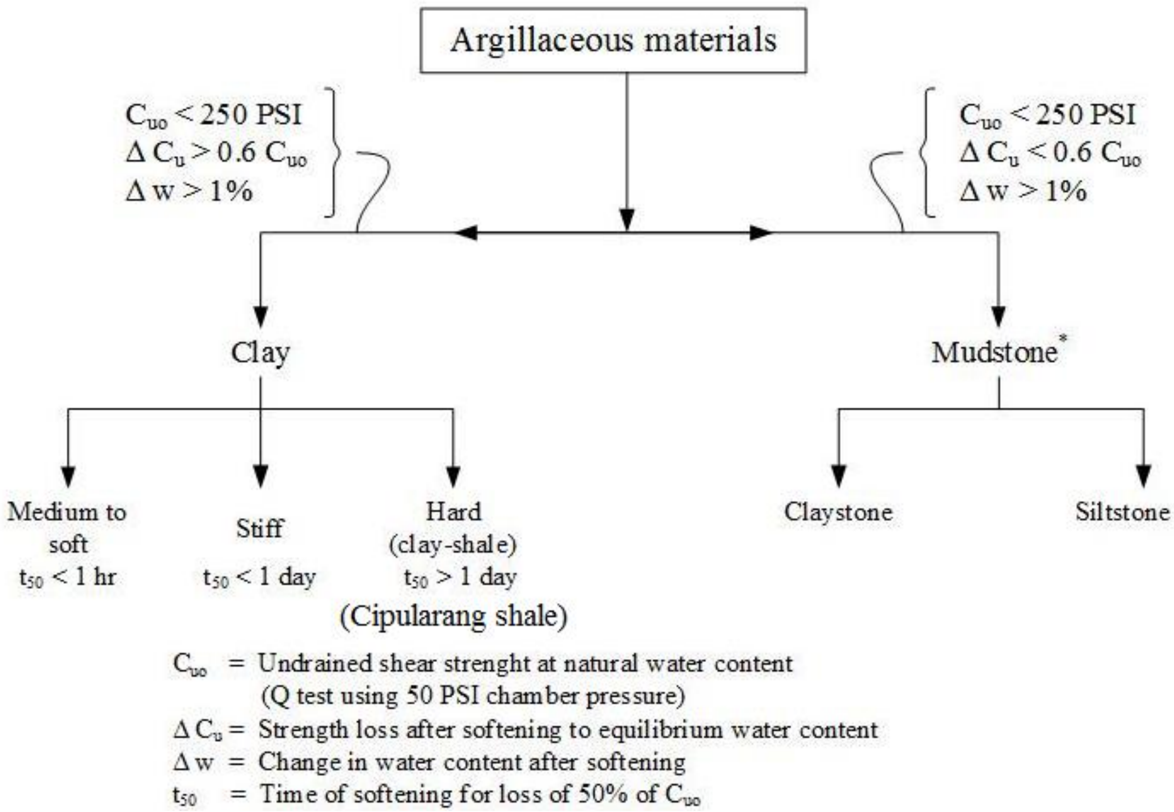

Figure 7. Two parts of classification scheme based on minimum 50% clay sized particles (Morgenstern and Eigenbrod, 1974).

such as *"over-consolidated"* (Johnson, 1969; Fleming et al., 1970), and "stiff, fissured clay" (Chandler, 1970) to indicate weakly bonded shale. This inconsistency in terminology has been most pronounced for the argillaceous materials that are transitional between normally consolidated clays and intact shales. Attempts were made by some investigators (Mead, 1936; Philbrick, 1950) to account for the potential changes in material behavior with time. The influence of durability was considered and the term *"slaking"* is introduced in their classification schemes. This is based on correlations of material properties, such as moisture content, liquid limit, dry density, etc.

These authors informed that classification based on geotechnical is considered not only for clayshale behavior, but also grain size distribution, shear strength, overconsolidation ratio, and Atterberg limits. In the beginning, because time factor is not calculated, that classification is valid for all clays in normally consolidated (Terzaghi, 1936; Bjerrum, 1967; British Standard Institute Classification (BSI), 1957). However, for clay and shale in overconsolidated, these classification limits are assumed inadequate, so that in further development, classification has to consider durability factor. Morgenstern and Eigenbrod (1974) also suggested 2 (two) types of classification where they are focussed on alteration of undrained shear strength and water content after softening (Figure 7). By the value of uniaxial compressive strength of rock (q_u), Deere and Miller (1966) divided by 5 (five) classes for modulus variation started from the lowest to the highest stiffness (Figure 8). Based on the value of modulus, rock material is divided by 5 classes, that is, the lowest to the highest stiffness values.

Gamble classification (1971)

Gamble (1971) carried out extensive investigation on the durability of varieties of shale; he strongly recommended that these materials could best be classified on the basis of the relationship between a two cycle slake durability index and their plastic index. Gamble suggested that more work was needed in order to correlate laboratory results with field behavior, but no attempts were made to connect between his classification scheme and the pre-established terminology. Gamble (1971) conducted the research on shale durability from various location and consistency. By physical properties (water content, liquid

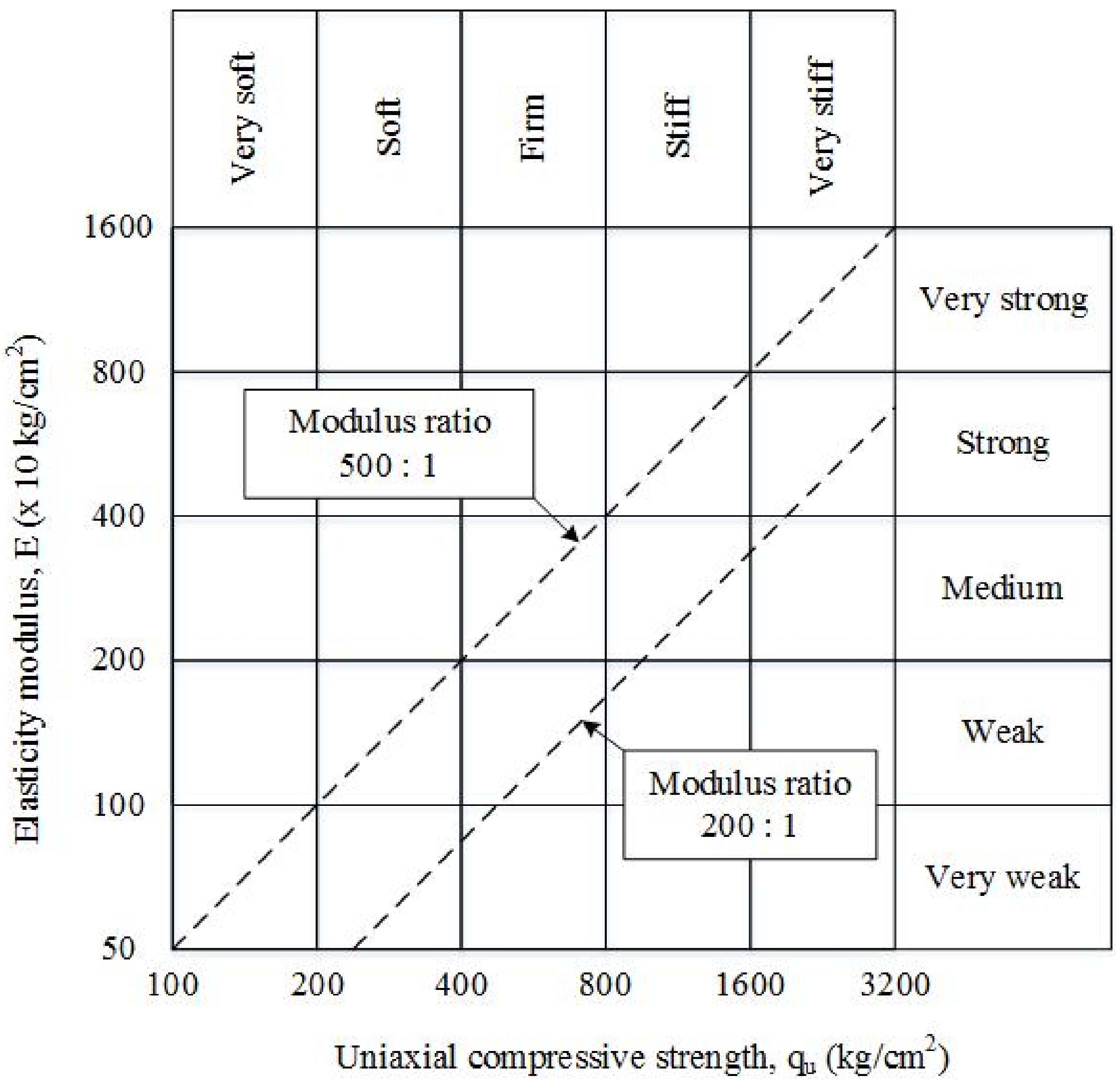

Figure 8. Rock classification (Deere and Miller, 1966).

limit, etc), shale classification is divided according to *slake durability (I_{d2})* index and plasticity index (PI). In this classification, it required Atterberg limits (LL and PL) and slake durability test as shown in Figure 9 (Gamble, 1971; Franklin and Chandra, 1972).

Deo classification (1972)

Based on the realization of the importance of shale deterioration, another classification was proposed by Deo (1972) that classified argillaceous materials according to their susceptibility to deterioration rather than the initial state of the material. Three tests, all of which measure shale durability (that is, slaking, slake durability, and sulfate soundness), were performed on various shales from Paleozoic deposits in Indiana. Using indices derived from these three tests, Deo categorized shale deposits into soil-like shale, two types of intermediate shale, and rock-like shale. According to Deo (1972), shale is divided by 4 (four) types, such as: (1) soil-like shale; (2) Intermediate-2 shale; (3) Intermediate-1 shale; and (4) rock-like shale with some criterias based on I_d values from index durability test and/or sulfate soundness index (I_m). Deo (1972) suggested to perform one cycle of slaking test. It would be soil-like if the test result indicated a weak shale condition (Figure 10). If slaking in one cycle is not completed, then it can be continued with one of the tests or combination slake durability test (one cycle) for dry sample, slake durability test (one cycle) for soaked sample, or modified soundness test.

In principle, modified of soundness test is same as

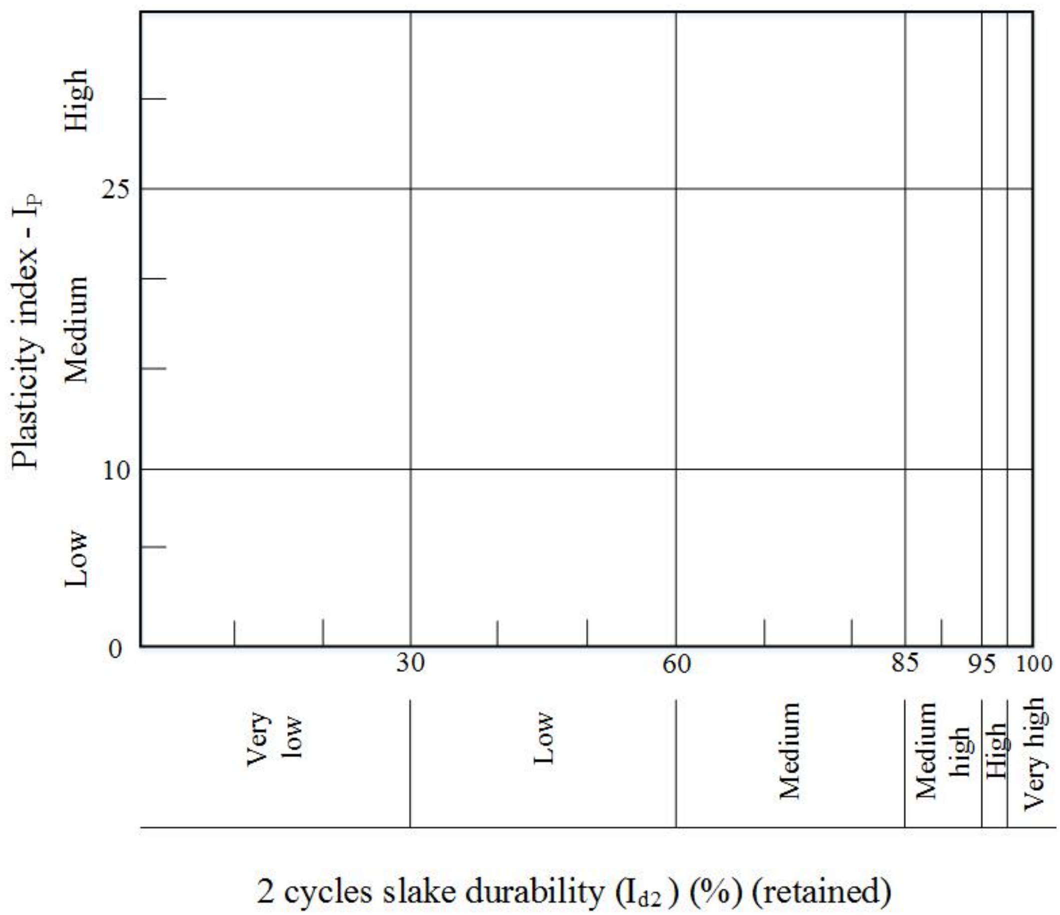

Figure 9. Gamble's geotechnical classification (Gamble, 1971).

slake durability test. Modification refers to use the sodium sulphate or magnesium sulphate solution where dry sample previously soaked into this solution. Percentage of retained on 3/8" (9.5 mm) sieve after slaking test finished is called *"sulphate soundness index."*

Morgenstern and Eigenbrod (1974)

A combination of earlier classification schemes based on initial properties and classification schemes based on durability was first attempted by Morgenstern and Eigenbrod (1974) who presented two classification schemes (Figures 7 and 11 and Table 2); one based entirely on the slaking characteristics (that is, the rate of slaking versus the amount of slaking), and a more significant scheme that included undrained shear strength, strength loss after softening, changes of water content after softening, and the time of softening. Although, it was required that the scheme emphasizes the influence of softening on strength and water content, the scheme first stipulated three potentially conflicting properties: (a) undrained shear strength, (b) the degree of strength loss after softening, and (c) the degree of changes in water content after softening.

These properties are given conditional values prior to dividing the argillaceous material into either soil or rock, and the classification is based only on these conditional values. After this division, slaking characteristics are used to determine if any of the soil like materials is clay shales. According to this classification, a shale that could be classified as rock-like according to its initial strength characteristics, could also be classified as soil-like based on its response to softening. According to this scheme, Italian clay shale, although rock-like in initial strength, slakes completely to a soft mud with only one cycle of the slake durability test (Belviso et al., 1977). Other engineering materials are classified according to their engineering properties that they presently exhibit. Yet, a *"clay shale"* is unique not in its present properties, but rather in its potential for significant deterioration of these properties as a result of interactions with water. None of

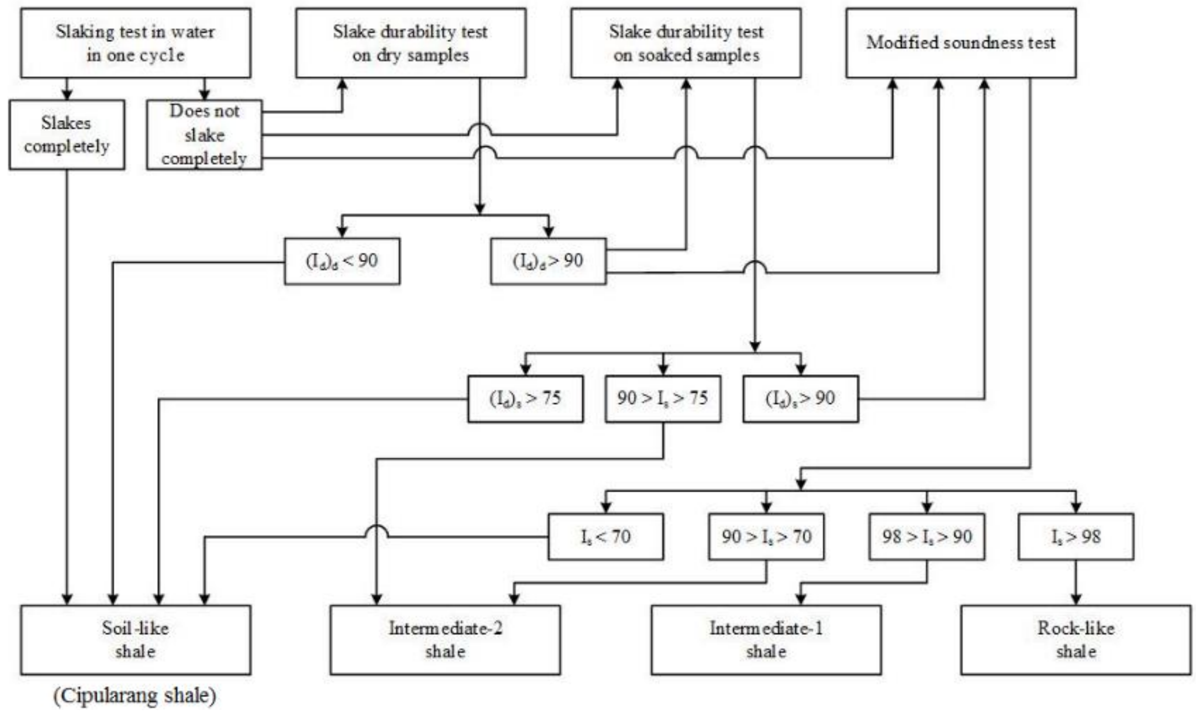

Figure 10. Deo's classification of shales (Deo, 1972).

the classification schemes to date have succeeded in recognising that. For instance, stiff clay, such as the London clay; a clayey shale, such as the Pierre shale; or a well bonded shale such as Ashfield shale, are terms that define these materials according to their present engineering properties such as plasticity, slaking, and softening. However, based on the method of Morgenstern and Eigenbrod (1974), all of them regardless of the rate of deterioration can be further classified as *"clayshale".*

Morgenstern and Eigenbrod (1974) divided by 2 (two), such as: soil and rock. If S_u < 18 kg/cm^2 then they like soil behavior and classified as clay. Vice versa, for category S_u > 18 kg/cm^2; this is called rock. In this classification, clayshale is a *"transition material between soil and rock."* For this category, clayshale is same as hard clay or rock with fissility signed by a weak zone.

Shale durability is based on slaking rate and total strength reduction reaching 50% occured by soaked effect. Caused by soaked, shale tends to absorb water and induces the softening reaching to its liquid limit.

Total of disturbed slaking relates with the liquid limit. This matter relates with the material potency to absorb water. Material with high liquid limit would change relatively caused by slaking if they are compared to the material with low liquid limit values. Slaking rate depends on liquidity index (LI) conducted by the sample soaked during 2 h.

Slaking durability is determined by liquid limit value and divided by 5 from low to the highest levels.

Final results on this classification are information of slaking rate and durability of rocks. The method requires the number of samples to be relatively more than the previous two methods; advantages of Morgenstern and Eigenbrod is the ability to determine slaking rate besides the durability of rocks.

Franklin (1981)

Franklin classification (1981) includes slake durability index (I_{d2}), point load index ($I_{s(50)}$), and plasticity index factors to obtain shale rating. Point load index is used to classify the durable shale on the limit I_{d2} > 80%, and plasticity index is applied for I_{d2} < 80% (signed by 'red line') (Figure 12).

Shale rating is the value starting from 0 (zero) to 9 (nine), where 0 (zero) shows the lowest durability and 9 (nine) indicates the highest durability.

METHODOLOGY

Sampling

Disturbed and undisturbed sampling from 2 (two) borlogs was carried out at Cipularang Highway near 97 + 000 and 97 + 300 km (Figure 4) from depth of 2.0 to 12.0 m, using manual drilling tool

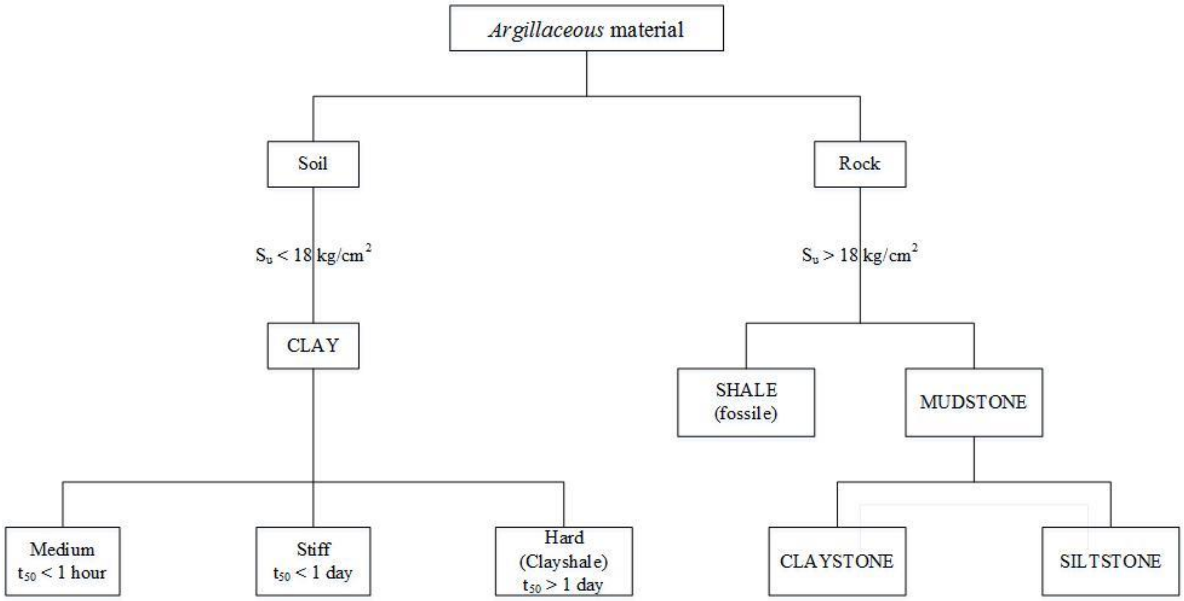

Figure 11. Geotechnical classification (Morgenstern and Eigenbrod, 1974).

Table 2. Morgenstern and Eigenbrod's geotechnical classification of shales (Morgenstern and Eigenbrod, 1974).

w_l = Liquid limit w_s = Max water content due to slaking I_L = Liquidity index ΔI_{LI} = Change in liquidity index		Amount of slaking $w_s = w_l$				
		Very low VL $w_L < 20$	Low L w_L between 20 & 50	Medium M w_L between 50 & 90	High H w_L between 90 & 140	Very high VH $w_L < 140$
Rate of slaking: $\Delta I_{LI} - I_{LI} - I_{L0}$ (2 hr water immersion)	Slow, S $\Delta I_L < 0.75$	VL S	L S	M S	H S	VH S
	Fast, F $0.75 < \Delta I_L < 1.25$	VL F	L F	M F	H F	VH F
	Very fast, VF $\Delta I_L < 1.25$	VL VF	L VF	M VF	H VF	VH VF

and drilling machine.

Geological and geotechnical classification

According to geological classification from Wenworth (1922), Ingram (1953), Folk (1968), and Underwood (1967), based gradation curve as shown as in Figure 13 is dominated by silt and clay sizes. This material can be identified as *"clayshale"* with *soil-like* behavior.

Using rocks classification by Deere and Miller (1966), clayshale Cipularang (97 + 000 km) from Jakarta based on modulus elasticity (E) and uniaxial compressive strength (q_u) can be classified as *"very soft rock"* with the lowest stiffness, where E value is lower

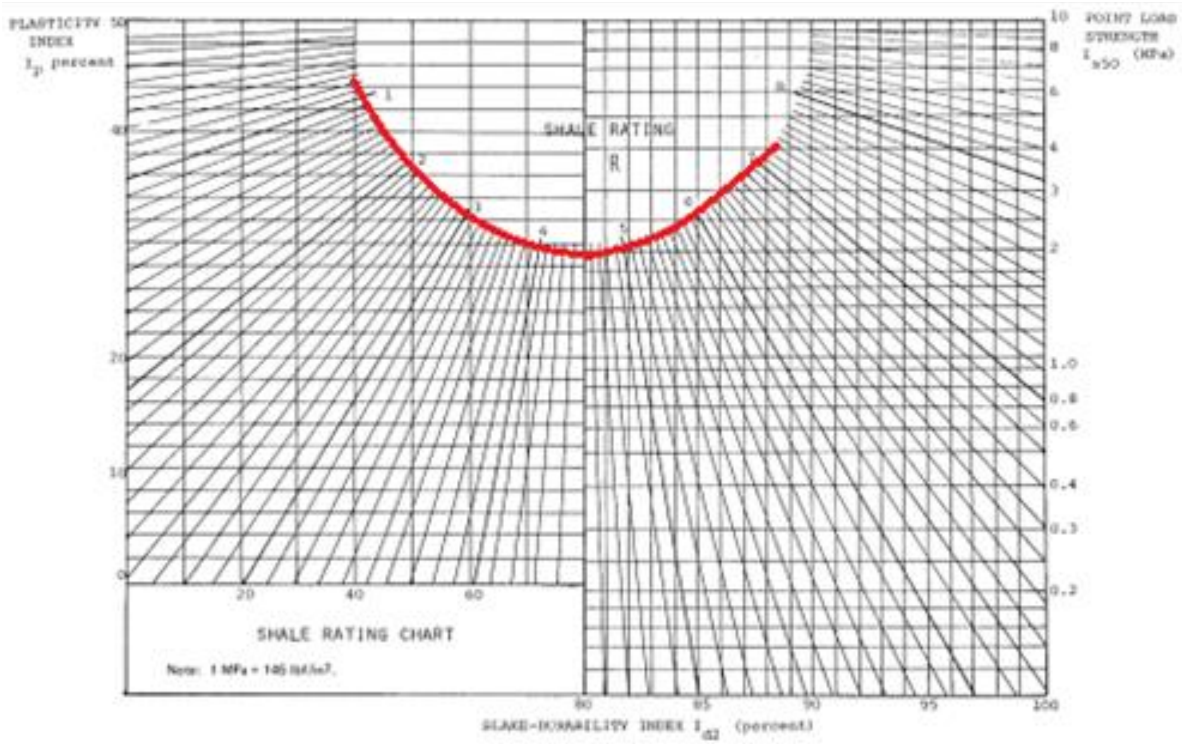

Figure 12. Durability rating of shale (Franklin, 1981).

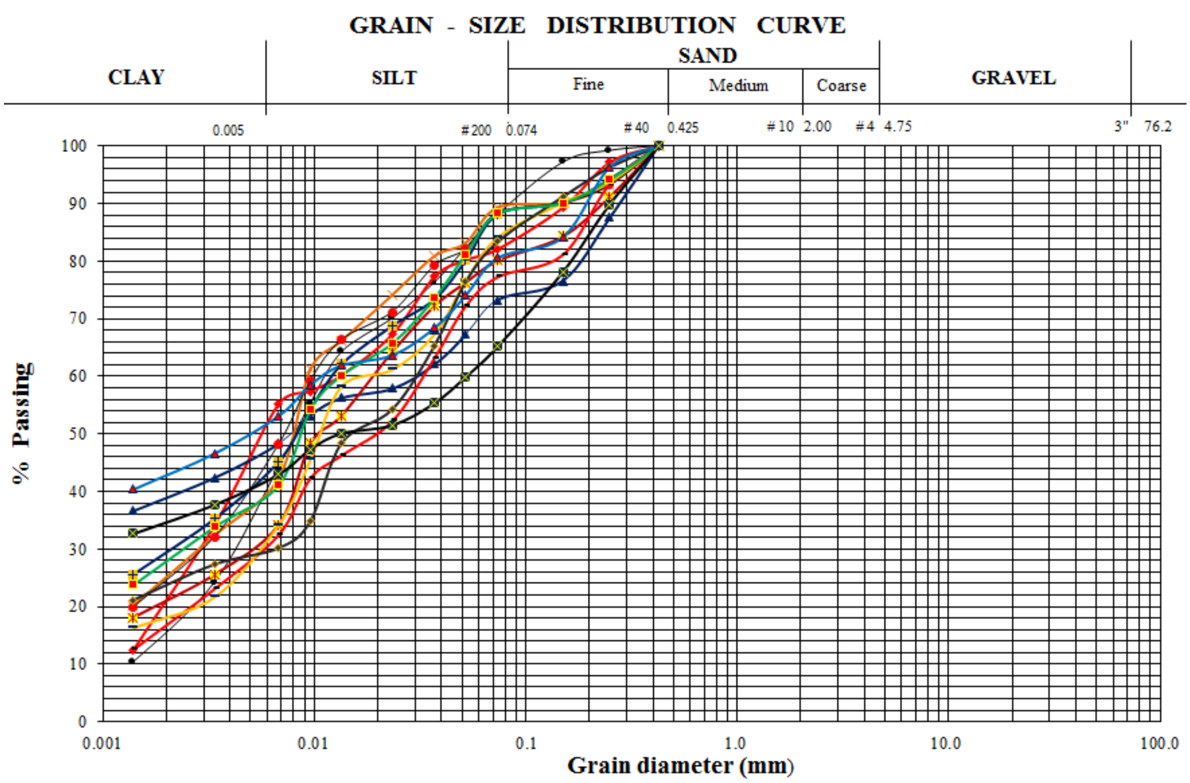

Figure 13. Grain size distribution.

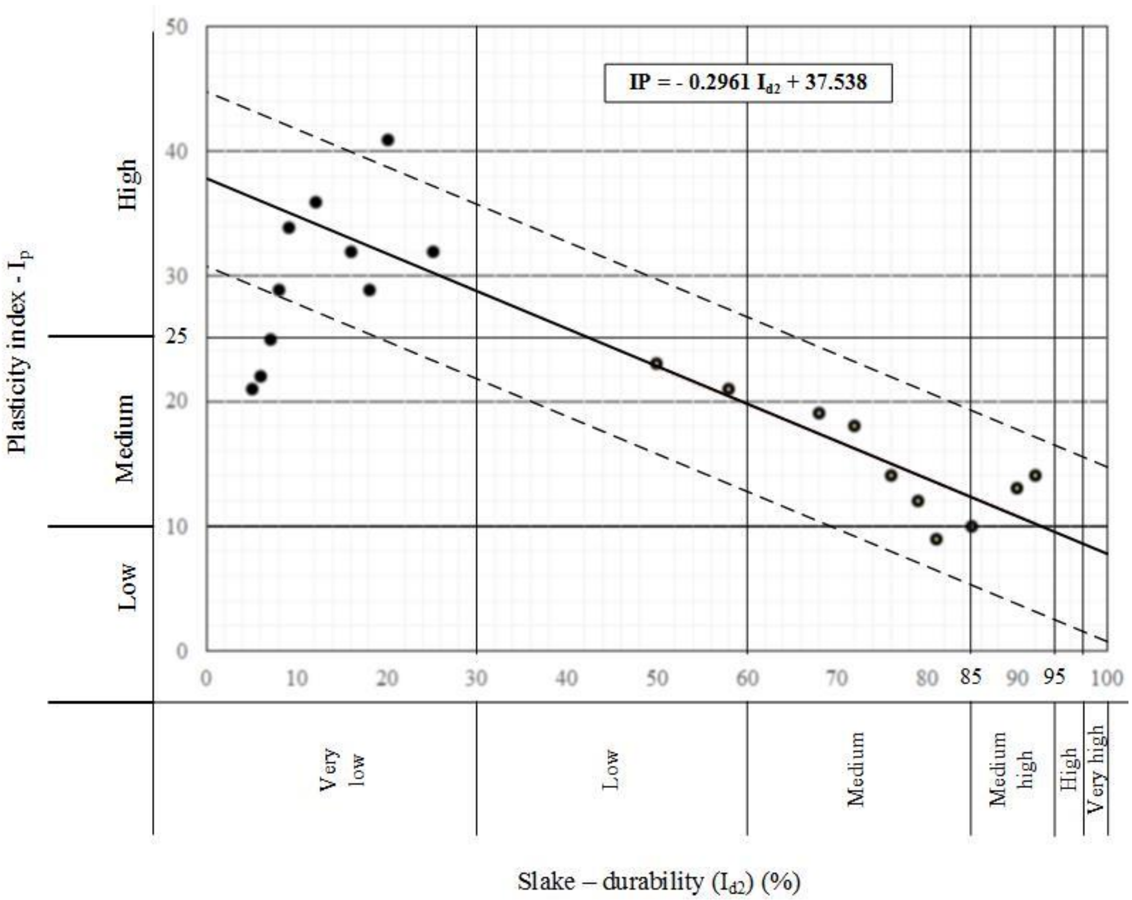

Figure 14. Clayshale classification of Cipularang Highway based on Gamble (1971).

than 100,000 kg/cm₂ and q_u value lower than 200 kg/cm^2.

RESEARCH RESULTS

Geotechnical classification is more focussed on material properties of clayshale based on durability level and behavior tendency of shale (*soil-like shale* or *rock-like shale*). Each classification is analyzed as the following.

Gamble (1971)

Based on durability classification by Gamble (1971), clayshale is fairly spread (Figure 14). In general, Cipularang clayshale is included in the group of low durability with plasticity medium to high and medium to high durability with low to medium plasticity.

Durability (I_{d2}) of rock data is lower than 30%; it was difficult to perform an uniaxial compression test because sample is fragile and collapse in test preparation. It was the same with the point load test where samples were damaged when the test started. Figure 14 shows a specific pattern: if plasticity index increases, durability of rock decreases.

Deo (1972)

Based on total sample and slaking-durability tests (I_{d1} and I_{d2}), Deo's classification is used to predict the bahavior of shale. For this case, shale tends to include in the analysis of soil behavior. This is consistent with geological classification from Underwood (1967) where it was shown that shale exists in *soil-like shale* behavior.

Morgenstern and Eigenbrod (1974)

Based on the result of triaxial test, rock properties are fissility signed by a weak zone, where the weak section indicates shale fragments with undrained shear strength (S_u) between 20 and 30 kg/cm^2. With the assumption of S_u value obtained by uniaxial compression test equals ½

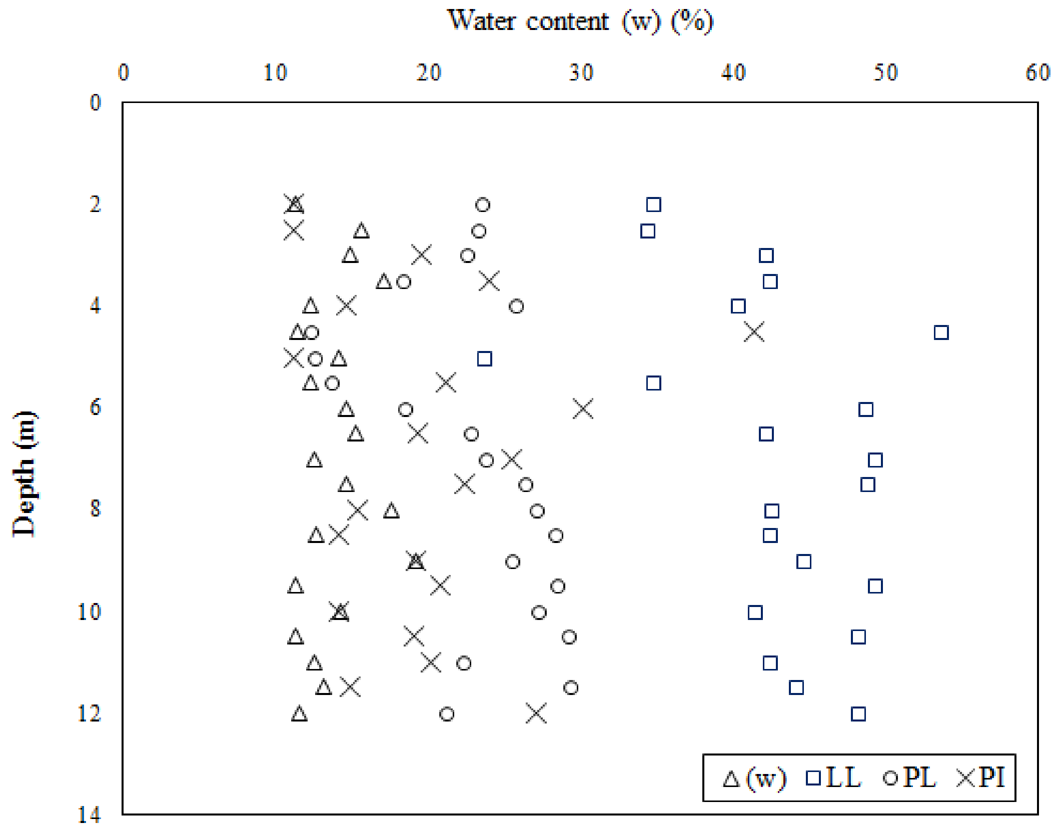

Figure 15. Profile of water content and Atterberg limits.

q_u, so that S_u value would be tried to classify with average of S_u values more than 18 kg/cm^2 (Figure 11) and according to Morgenstern and Eigenbrod it could be categorized as *clayshale*.

Franklin (1981)

Based on shale rating by Franklin (1981) in Figure 12, results show that shale rating exists in the range between 0.60 and 7.40. According to this rating, category of shale-rock indicates low to high durability.

Value of q_u from clayshale ranges between 17 and 172 kg/cm^2 (Deere and Miller, 1966), for rock with q_u < 200 kg/cm^2 could be categorized as very soft rock (Table 1).

Based on geotechnical classification by Gamble (1971), Deo (1972), Morgenstern and Eigenbrod (1974), and Franklin (1981), it could be concluded that shale behavior is classified as *soil-like shale* with variation of durability level from very low to high (Table 2).

From elasticity modulus (E) value ranges between 1559 to 10400 kg/cm^2; this material could be grouped into very low stiffness (Deere and Miller, 1966). Therefore, Cipularang clayshale could be classified as a very soft rock with very low stiffness (Figure 8) (Deere and Miller, 1966).

Water content and Atterberg limits

Figure 15 shows that natural water content condition exists below plastic limit. Average of natural water content (w_n) is 16% with mean of plastic limit (PL) equals 23% and liquid limit (LL) equals 43%. Average of natural water content is more than 7% when they are compared with PL.

According to Seed and Woodward (1964), Sowers and Sowers (1970), Gamble (1971), and Mitchell (1993) and Soga (1994), although each differ in limitation on the PI values to swelling potential, Cipularang shale shows PI values <30%; the swelling potential of clay mineral is low.

Void ratio and total vertical stress

Void ration (e) of Cipularang clayshale varies between 0.12 and 0.41. According to Attewell and Farmer (1976) and Winterkorn and Fang (1975), void ratio of shale is in

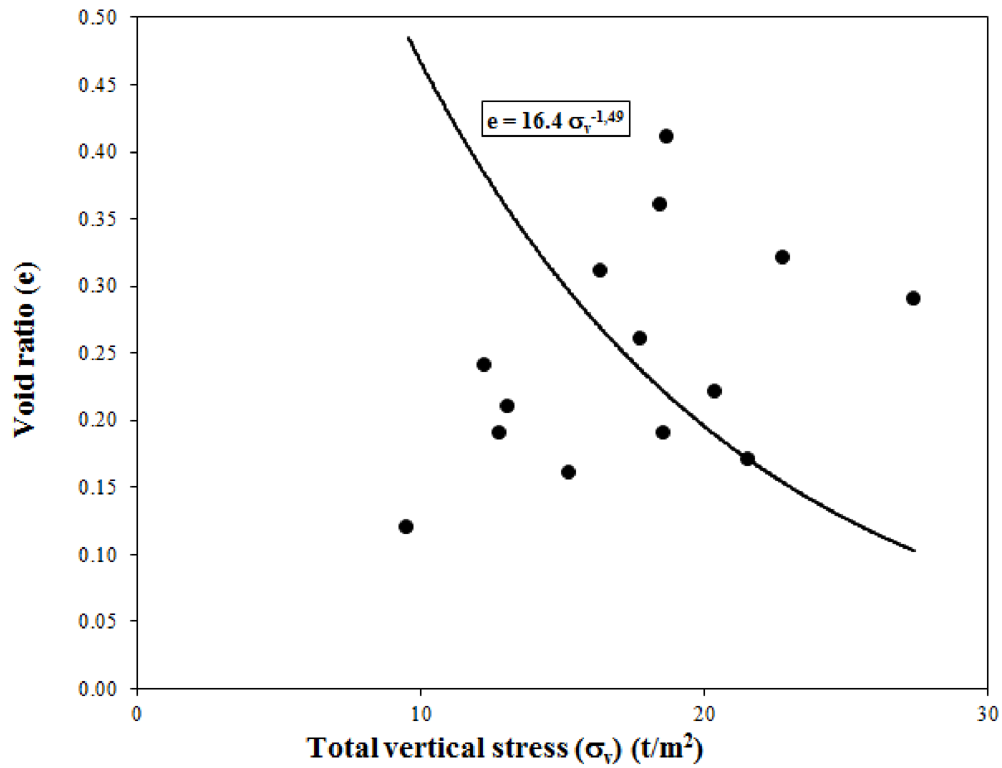

Figure 16. Relation between total vertical stress and void ratio.

the range of 0.11 to 0.43. Some studies from Clark (1966) and Brace and Riley (1972) concluded that void ratio for shale is between 0.08 and 0.50. Furthermore, relationship between void ratio and total vertical stress, where the total vertical stress increases, void ratio tends to decrease under the overburden pressure working on the deposit (Figure 16).

Clay mineral content

Based on the results of index properties using Cassagrande Plasticity Chart indicates this shale dominated by clay mineral with low plasticity (CL). Activity (A) values between 0.13 and 0.5 and clay content between 32 and 65% from existing laboratory results. So, actually deposit shale is difficult to change into a liquid state in rainy season. In natural state, mean of G_s value of 2.64 closes to clay soil. Most results indicate that G_s value at 97 + 000 km is exactly not similar with 96 + 600 km.

Clay content and liquid limit

There is correlation between liquid limit (LL) and percentage of clay as shown in Figure 17. From some results, increasing clay content shows the increasing liquid limit (LL) value. The relationship could be indicated by LL = 0.72% Clay + 3.24.

Liquid limit (LL) with plasticity index (PI) is illustrated in Figure 18, where plasticity index for Cipularang clayshale is predicted using equation PI = 0.74 (LL – 16.2). The equation indicates similarity geological characteristic because the line of equation is more or less close to the “A” line Plasticity Chart for USCS or Unified Soil Classification System (Casagrande, 1948; Howard, 1977).

Weathering and slake durability

Laboratory works of slake durability test (Franklin and Chandra, 1972; Deo, 1972; Moriwaki, 1975) was carried out using ASTM D 4644 – 04. Typical process of slake durability test results are presented in Figure 19. Number of cycles for all slake durability test conducted ranges from 1 to 4. Sample of weathered clay shale is decayed in maximum of 2 (two) cycles.

In Figure 20, weathering process may strongly influence durability sample. Sample weathered clayshale was left exposed in the air during 1 (one) month and

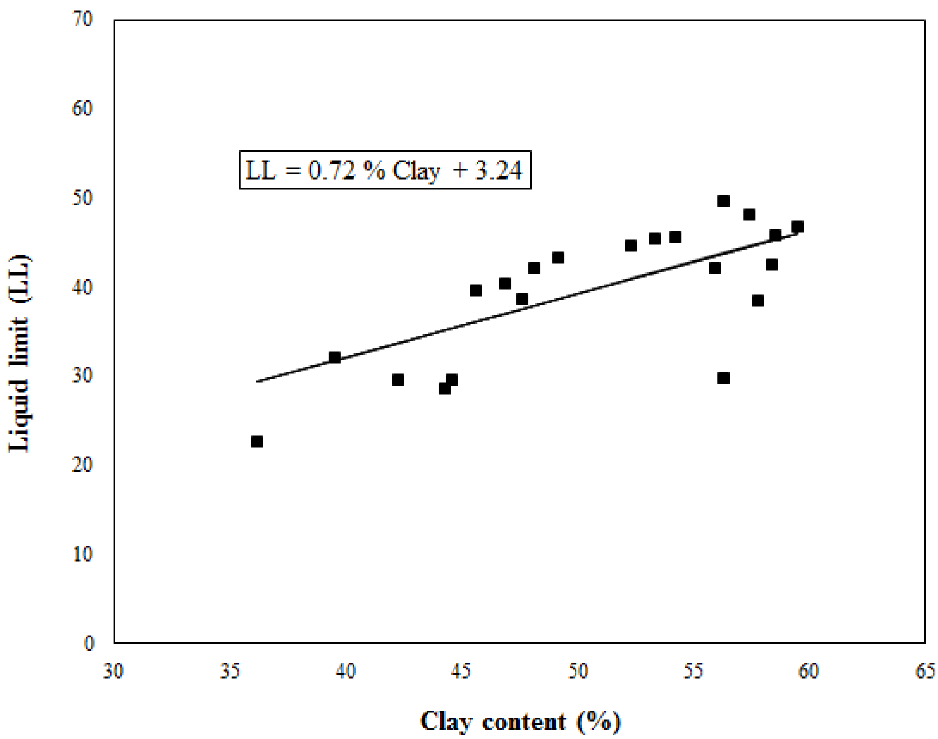

Figure 17. Relation between liquid limit (LL) and percentage of clay content.

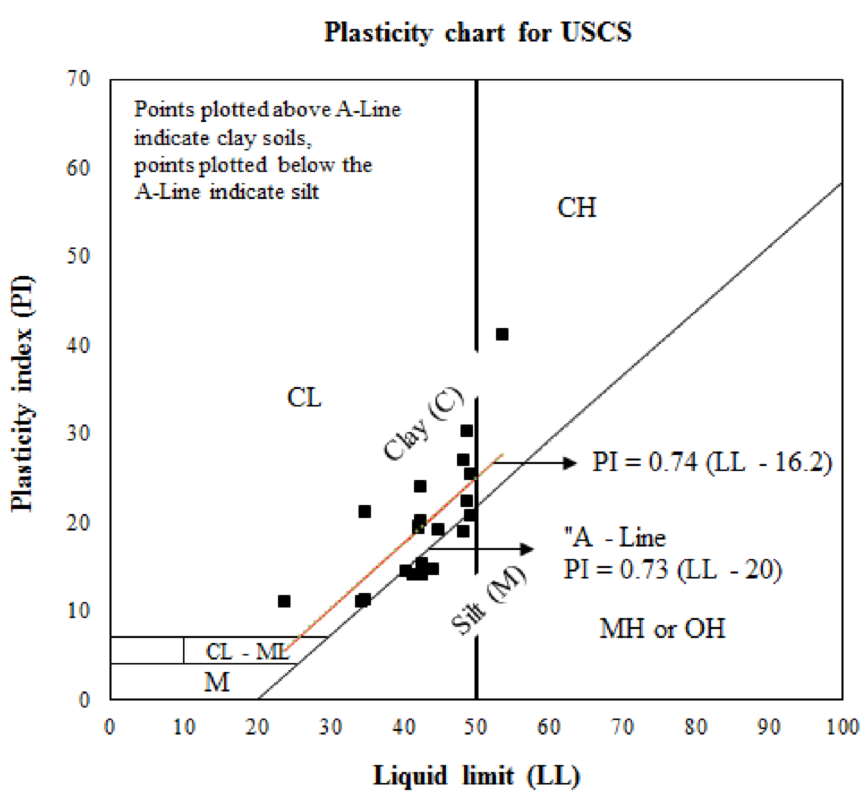

Figure 18. Relation between liquid limit (LL) and plasticity index (PI).

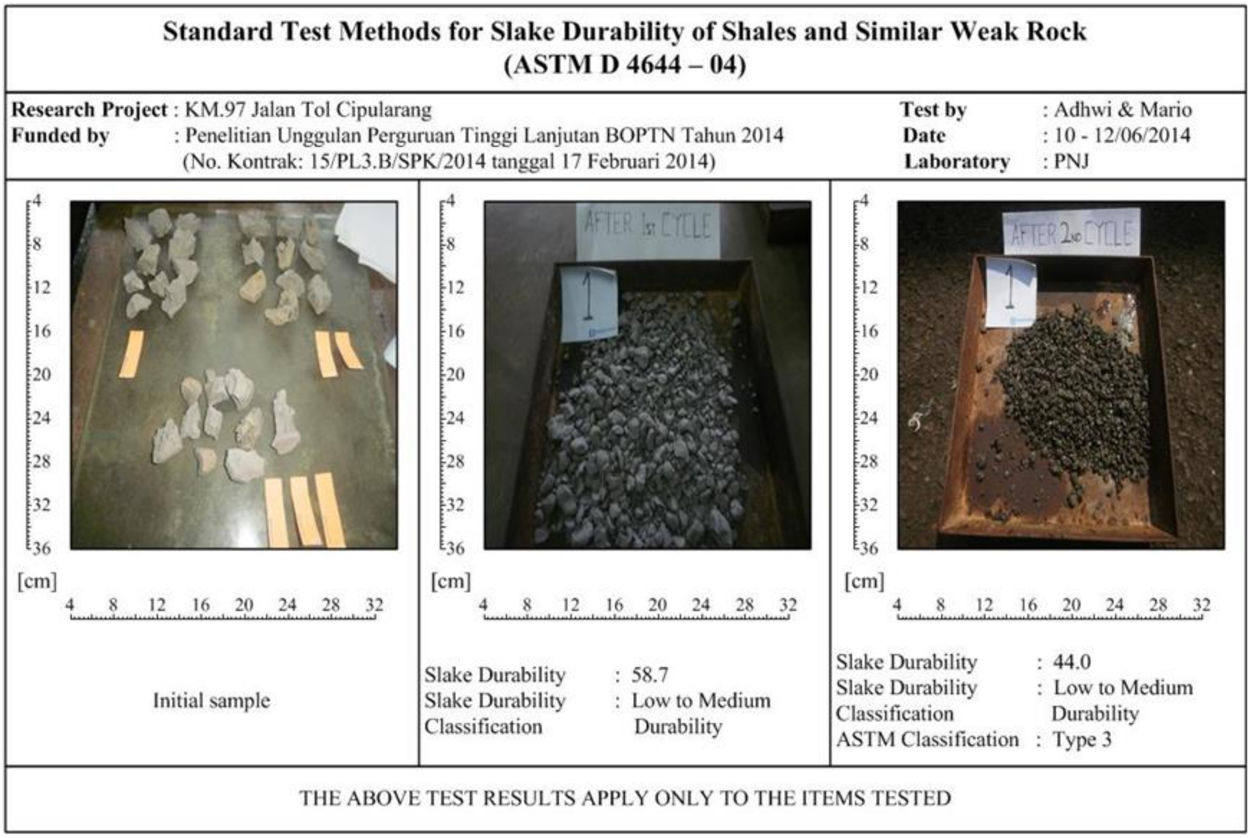

Figure 19. Typical process of slake durability test.

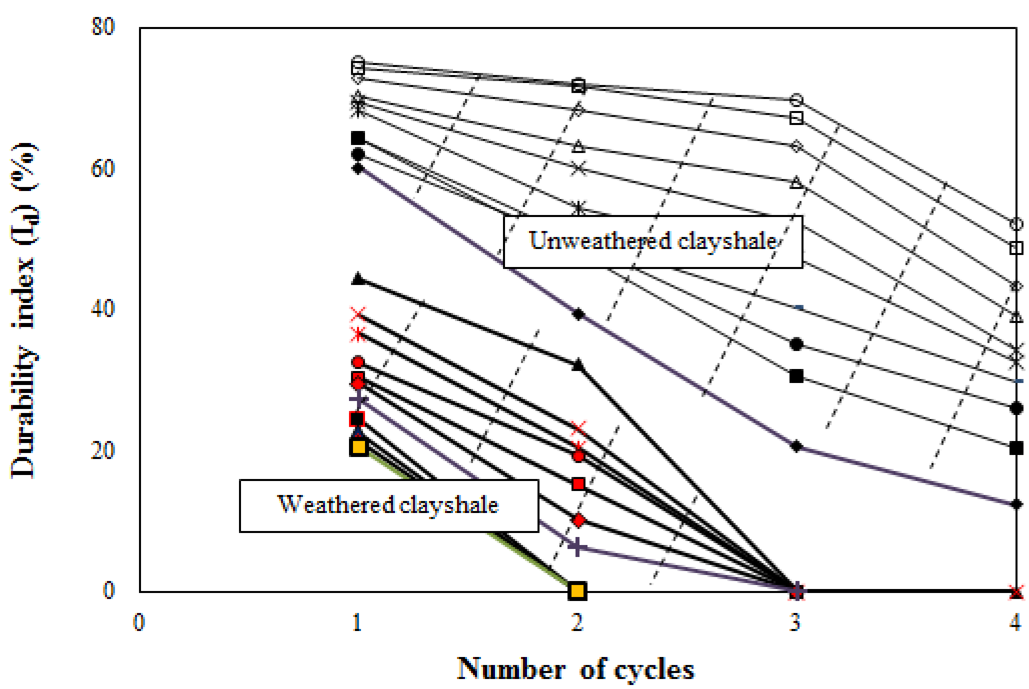

Figure 20. Relation between number of cycles (slake durability test) and durability index (I_d).

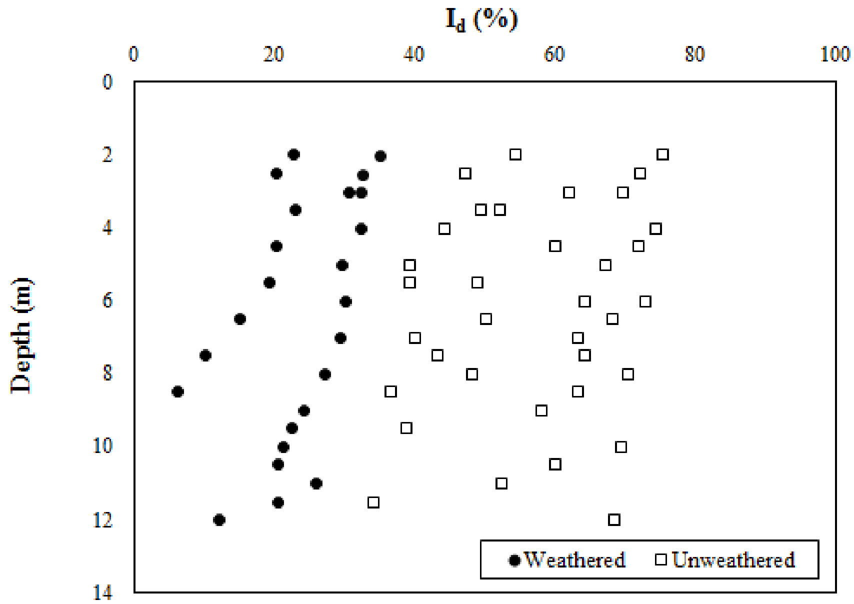

Figure 21. Relation between I_{d2} and persentage of clay content with depth.

indicated significantly reduction of durability. Reduction of durability is determined by $I_{d(2)}$ value which may reach 50% durability of unwethered clayshale.

In Figure 20, the relationship between I_{d2} and sample depth shows the unweathered clayshale at the lower limit which durability may increase with depth. Weathered clayshale does not have a specific pattern. Figure 21 shows clay content tends to decrease with depth. Percentage of clay content of weathered clayshale relatively is higher than unweathered clayshale. This may show that weathering level could be defined by more clay content of clayshale. In higher weathering, the higher percentage of clay contains (Henkel, 1982). Clay content on weathered clayshale may tend to increase with depth caused by the weathering process and higher disturbance than unweathered clayshale (Figure 22). Slaking effect may influence durability of clayshale.

Liquid limit and slake durability index

Correlation was obtained between liquid limit and $I_{d(2)}$ weathered clayshale (Figure 23). Curve indicates information if liquid limit is more enlarge, $I_{d(2)}$ value diminishes with equation: $I_{d(2)} = -0.32\ LL + 34.51$.

Collapsible potential

Collapsible soils are relatively dry; they are low density soils which undergo a decrease in volume when they become wet for the first time since deposition. This decrease in volume normally occurs without any increase in applied pressure (Owens and Rollins, 1990). Soil collapse is usually associated with human activities such as construction of road or highway, or disposal of waste water that introduce water into a relatively dry environment. Although soil collapse is generally not life threatening, it can cause severe damage to road, drainage system, etc. (Prokopovich, 1984).

Gibbs and Bara (1962) have used a plot of dry density and liquid limit as a criteria for predicting soil collapse (Figure 24). Proposed criterion of Clevenger (1958) for collapsibility evaluation is based on the soil dry density, especially for the soil dry density is lesser than 1.28 g/cm^3 then the soil will collapse after minor water content change. On the other hand, if the soil density is more than 1.44 g/cm^3, then the lesser collapse settlement could be expected. For medium range of soil density, the medium collapse settlement could be evaluated.

Results shown in Figure 24 indicate that clayshale exists between collapsible and noncollapsible zones; it could be estimated that they are plotted in “intermediate zone of collapsible potential.” Alteration of clayshale behavior would occur when clayshale directly related to air and water at the time of stripping and disposal works for the upper layer (Figure 25). For this matter, clayshale is unlikely to collapse when they are not disturbed which could change their properties.

Based on the previous study, Cipularang clayshale would collapse caused by a loss of dry strength in the

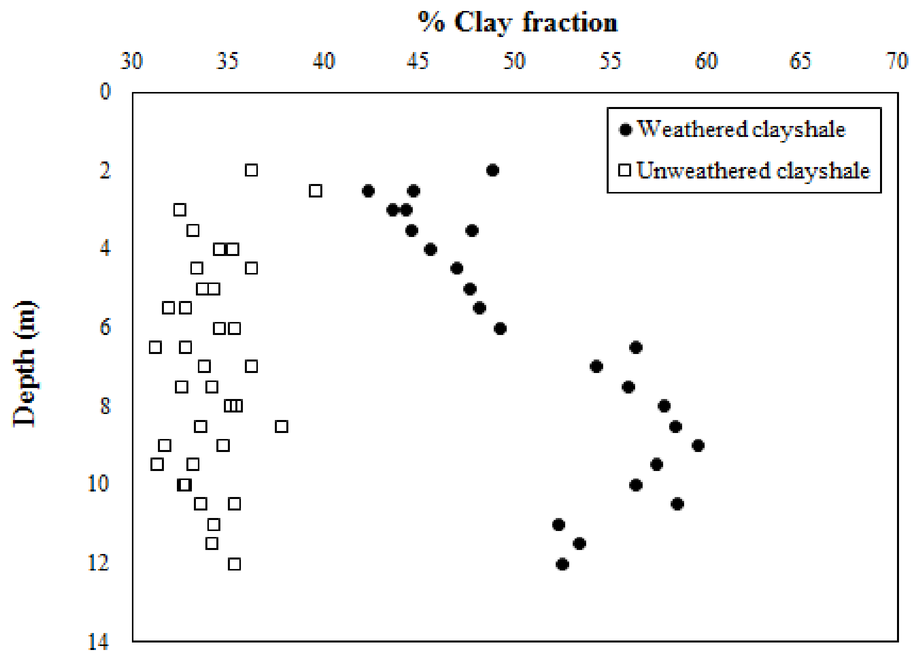

Figure 22. Relation between percentage of clay fraction and with depth.

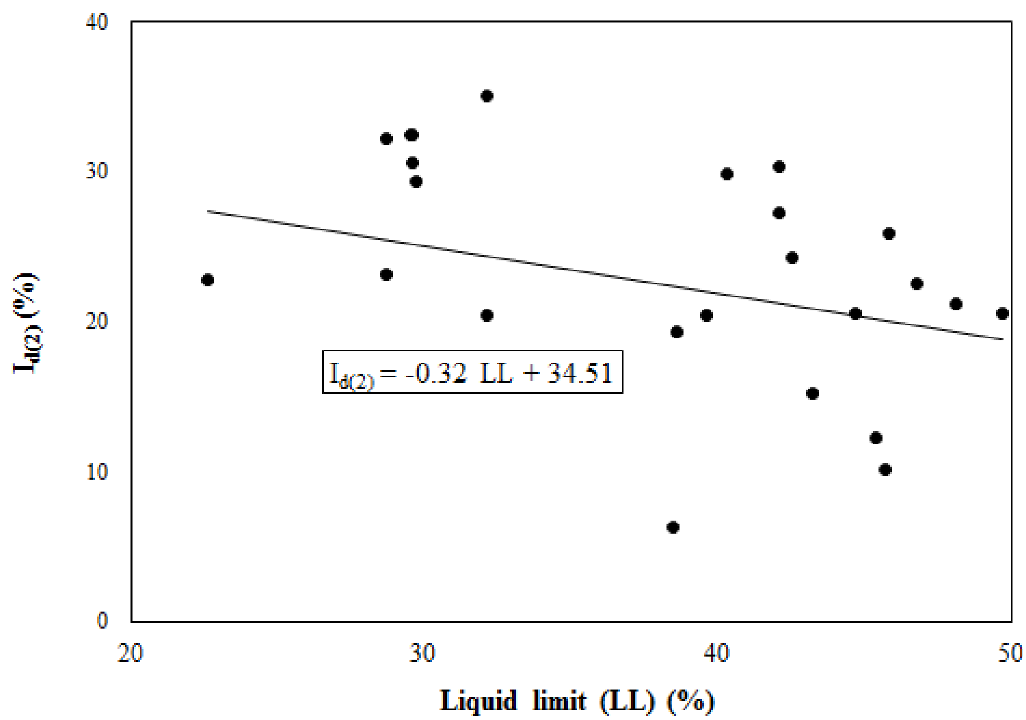

Figure 23. Relation between liquid limit (LL) and slake durability index ($I_{d(2)}$).

soils. A complete loss of dry strength occurs when the soil is saturated to the liquid limit. If the volume of water corresponding to the liquid limit stage is larger than the natural porosity, the material under normal conditions

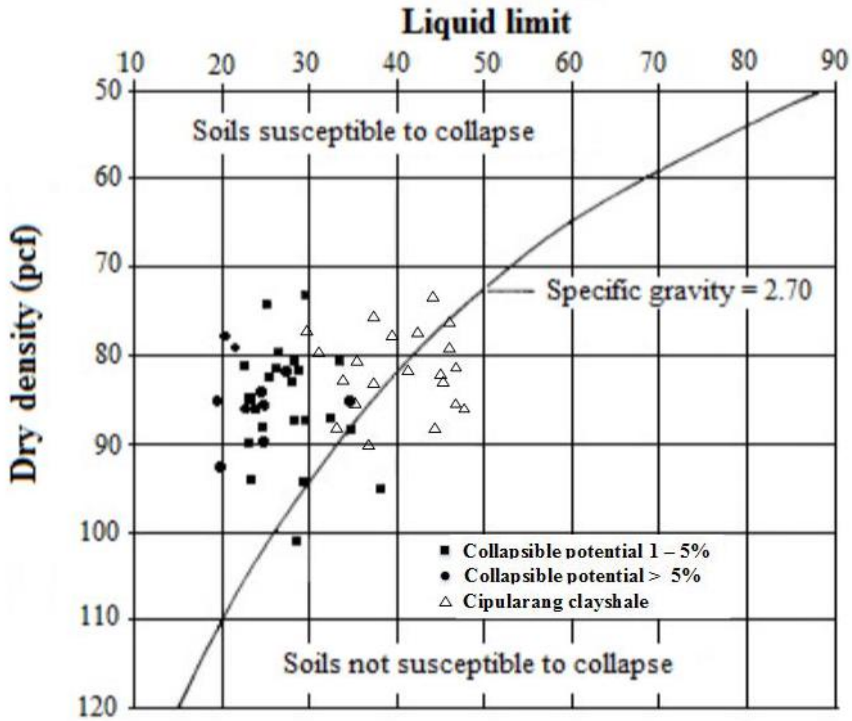

Figure 24. Susceptibility to collapse (Gibbs and Bara, 1962).

Figure 25. Stripping and disposal works for the upper layer (Jasa Marga (Persero), Tbk, 2003).

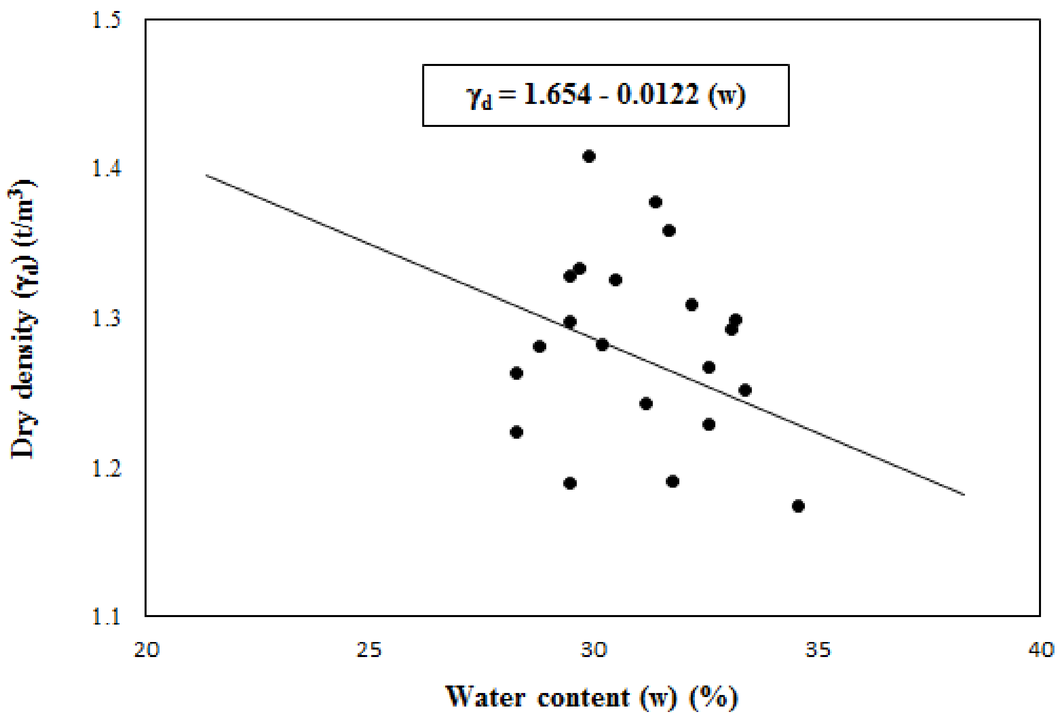

Figure 26. Relation between dry density (γ_d) and water content (w).

cannot be saturated to the liquid-limit. Therefore, it cannot completely lose its dry strength and is not considered collapsible. If the volume of the natural porosity exceeds the volume of water required to reach the liquid limit, the soil may be "liquified" and may be subject to collapse (Prokopovich, 1984). Soil densities plotted above the line shown in Figure 24 are in a loose condition and will have a moisture content greater than the liquid limit. Therefore they will be susceptible to collapse. Soils plotted below the line are presumably not susceptible to collapse.

Prokopovich (1984) argues that theaforementioned method is invalid because collapse can occur when the moisture content of the soil is well below the liquid limit, and that the relative strength and other properties vary between the undisturbed and remolded clays. Samples with a collapse potential greater than 1.0% were plotted in Figure 24. With Prokopovich's limitations in mind, it can be seen that there is generally a good correlation between the liquid-limit; dry density and the susceptibility to collapse for soils with a collapse potential from 1 to 5%. Figure 24 is a very good indicator of clayshale with a collapse potential greater than 5%.

Swelling potential

Activity (A) value obtained is between 0.23 and 0.65. This range is close to typical kaolinite and illite based on measurement from Skempton (1953), where Carter and Bentley (1991) found activity between 0.33 and 0.46 and Underwood (1967) detected 0.35 to 0.75.

From PL and LL values, it could be predicted that clayshale behavior may be influenced by mostly kaolinite. Low activity values show inactive clayshale and swelling potential is relatively small.

Dry density and natural water content

An increment of water content would reduce dry density as shown in Figure 26. This matter provides an information that if water absorption on clayshale with a certain amount of water content occurred, then it would be followed by reduction of density and volume increase.

Water content and compression strength

Based on uniaxial compression strength (UCS) test results, Cipularang clayshale has UCS values indicating strength rock classification of International Society for Rock Mechanics/ISRM (1978) from very low strength to low strength (Bieniawski, 1984). From Onodera (1970), clayshale from Cipularang could be classified as rock with highly weathered to weathered. Relationship

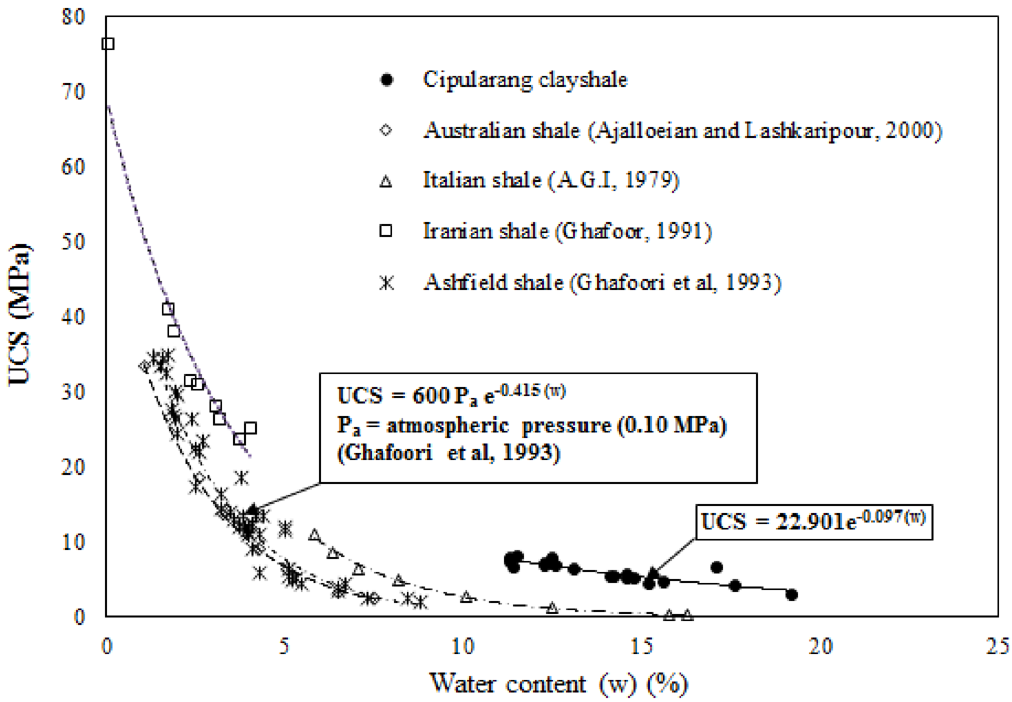

Figure 27. Relation between water content (w) and uniaxial compression strength (UCS).

between the uniaxial results and index properties of clayshale is shown in Figure 27. This matter indicates a specific trend where lower UCS or q_u value caused by water content increase.

When water content is expressed by liquidity index (LI), this relationship shows that smaller liquidity index could be higher UCS values. This is consistent with the fact naturally, that more brittle in the term of below plastic limit (PL) could be higher strength of material (Figure 28).

According to recommendation from ISRM (1985) with using point load test results and UCS, relationship between point load strength index ($I_s(50)$) and UCS could predicte Cipularang clayshale as UCS = 24.75 $I_{s(50)}$ (Figure 29). This correlation result (Figure 28) is slightly larger than that of Broch and Franklin (1972), and compared with Bieniawski's formula (1975) using 54 mm diameter (D) of thin-walled fixed-piston samplers and double-tube swivel type core barrels at Cipularang sampling area.

Modulus of elasticity

Laboratory results of uniaxial compression (UCS) test of Cipularang shale was around 123 to 6543 MPa. The results may be compared with laboratory dynamic (ultrasonic velocity) test used to determine wave velocity.

Based on investigation from Deere et al. (1967), the comparison between axial modulus (E_a) from the plate load test and modulus from the seismic in situ test ranges from 1/11 to 1 as a reduction factor. Empirical correlation from the plate loading test by Broms (1964) is k_s=1.67 E_{50} or k_s =4×103–1.6×104 q_u, k_s is coefficient of subgrade reaction.

Static Young's modulus from Bukit Sentul shale varies from 155.9 to 1040 MPa (Widjaja and Rahardjo, 2002; Widjaja, 2008). Values of static Young's modulus for North Sea Shales from various depths ranged from 800 to 12200 MPa (Horsrud et al., 1998), using undrained triaxial tests. These values fall within the aforementioned range.

As a comparison, Figure 30 shows $E_a/E_{ultrasonic}$ = 1/18.5 and $E_d/E_{ultrasonic}$ = 1/5.80. This difference can be caused by different types of test methods. Ultrasonic test uses a small strain, but uniaxial test uses a larger strain. These empirical equations are difficult to establish (Deere and Miller, 1966).

Compression strength and modulus

The increase of the uniaxial and triaxial compression strength is linear with increasing modulus. The relationship of E/q_u shown in Figure 31, the average value of E/q_u can be taken as: E_a/q_u = 50 (axial modulus) and

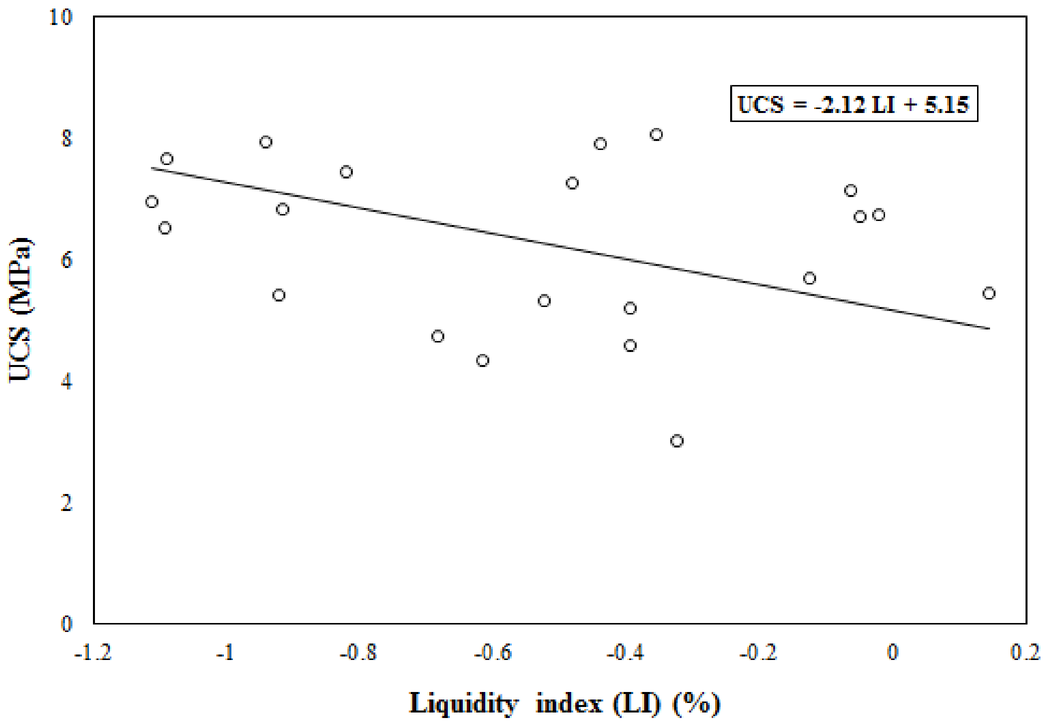

Figure 28. Relation between liquidity index (LI) and uniaxial compression strength (UCS).

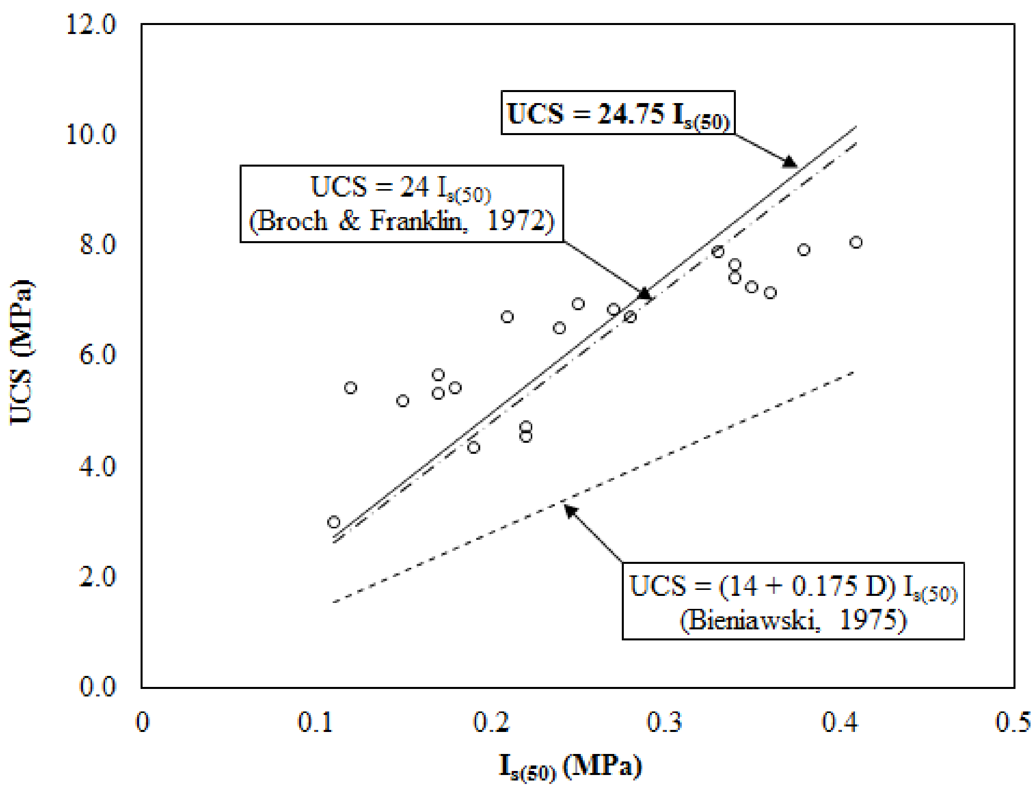

Figure 29. Relation between point load strength index ($I_{s(50)}$) and uniaxial compression strength (UCS).

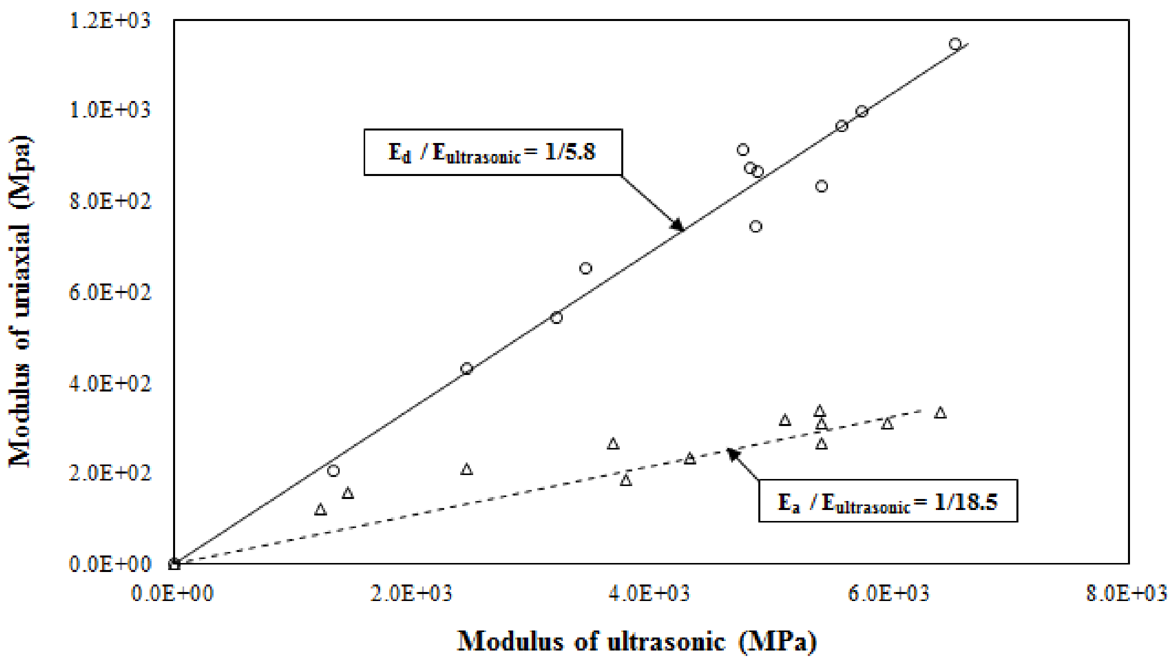

Figure 30. Relation between dynamic modulus by ultrasonic and static modulus by UCS tests.

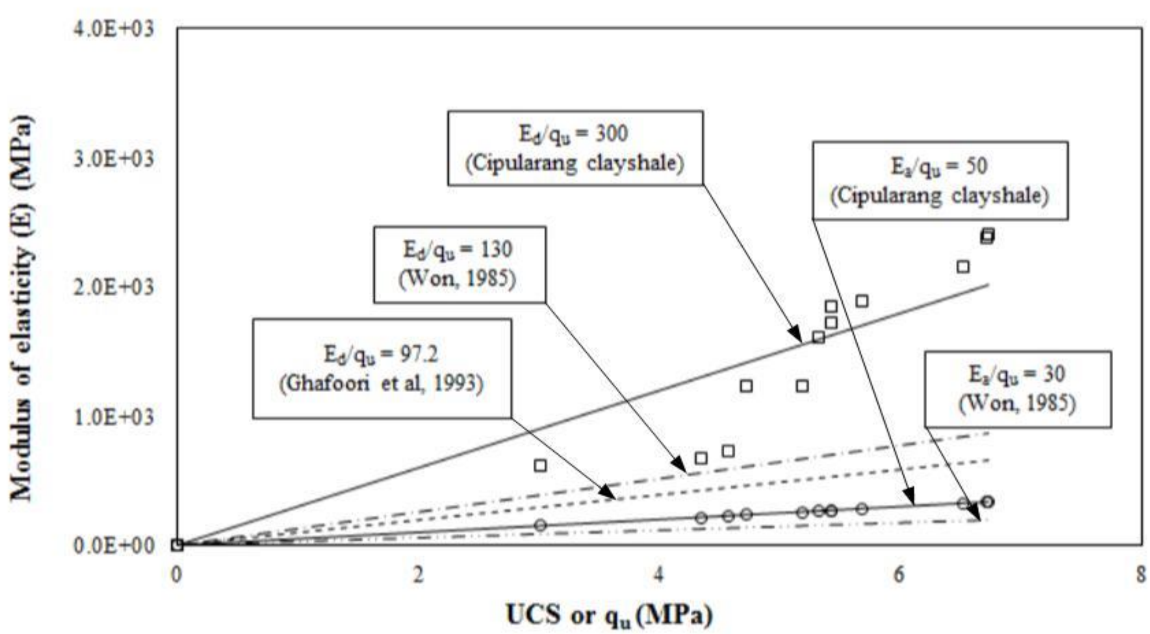

Figure 31. Relation between UCS and modulus.

E_d/q_u = 300 (diametrical modulus). The study of Won (1985), Ghafoori et al. (1993), Widjaja and Rahardjo (2002) and Widjaja (2008) show that the value of E/q_u of Cipularang clayshale closes enough to Ashfield shale and has a similar trend. The relationship showed that the modulus in diametrical direction is greater than modulus in an axial direction and also indicated the horizontal stiffness is greater. This shows that clayshale behaviour

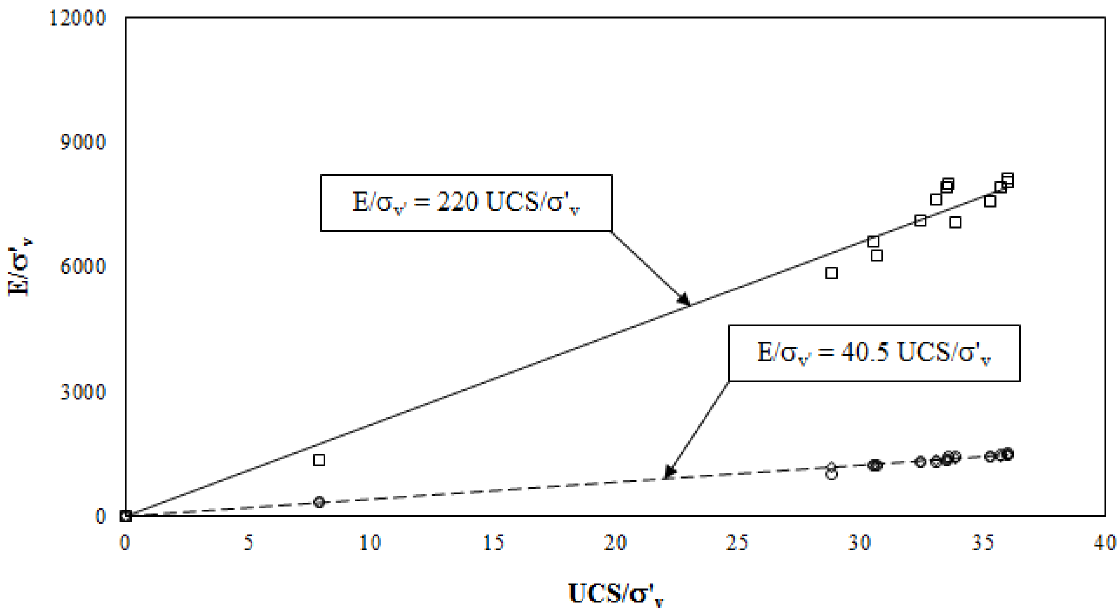

Figure 32. Normalized UCS and E value with respect to effective stress (σ'_v).

is anisotropic.

Normalized UCS/σ'_v and E/σ'_v in Fig. 32 provides equation E_d/σ'_v = 220 UCS/σ'_v and E_s/σ'_v = 40.5 UCS/σ'_v (Figure 32).

Shear wave velocity (V_s) and $I_{d(2)}$

Based on Japanese standard (Onodera, 1970), clayshale is divided by 2 (two) types, such as: highly weathered rock and slightly weathered rock related with durability from Gamble (1971) between low to high.

The relation between $I_{d(2)}$ and V_s may be retrieved at upper and lower bounds (Figure 33); from this figure it could be determined $I_{d(2)}$ using shear wave velocity (V_s). Furthermore, the figure could be applied to predict rock durability by using seismic refraction (down hole or cross hole test).

Field and laboratory shear strength

Undrained shear strength (S_u) based on results of uniaxial, pressuremeter and triaxial is shown in Figure 34. This figure indicates that average values of S_u increase by depth. Comparison of S_u values between uniaxial and pressuremeter relatively close at the same depth is around 1.1 to 1.5. Analysis shows that the test results of undrained shear strength are relatively higher than the uniaxial.

By using the results of soil modulus from uniaxial test (E_a), the value of soil modulus from pressuremeter test (E_p) at the same depth is around 1.4 to 1.9 higher than the soil modulus obtained by the uniaxial testing (E_a) (Figure 35). From some observations, the ratio of E_d/E_a is 1.6 to 2.1. Several studies from Hendron et al. (1970) indicated the result of ratio of E_d/E_a was 3.0. The analysis shows that the test results of modulus of pressuremeter are also relatively higher than the uniaxial test.

These differences may indicate that some problem during sample handling in the field; some differences of stress-strain mechanism during test; alternates or changes of water content when the test is performed; or some influences of slaking during drilling works.

The heavy overconsolidation of London Clay gives rise to high horizontal effective stresses, determining K_o values that are greater than 1. Skempton (1961) and Skempton and La Rochelle (1965) found that in the upper 10 m of the London Clay K_o varies between 2 and 2.5 and this value tends to decrease with increasing depth, falling to 1.5 at about 30 m depth. In Figure 36, the K_o profiles suggested by Bishop et al. (1965) and Hight et al. (2003) for Ashford Common, Heathrow Airport London and Cipularang shale are plotted simultaneously. Value of K_o on Cipularang clayshale determined by pressuremeter test indicates the K_o values between 1.6 and 2.5. Pattern of K_o values to depth also exhibits K_o values decrease with increasing depth. Peterson (1954) found the coefficient of earth pressure at rest could reach a value of 1.5. According to the study Skempton (1961) on the London Clay, K_o price varies with depth in the

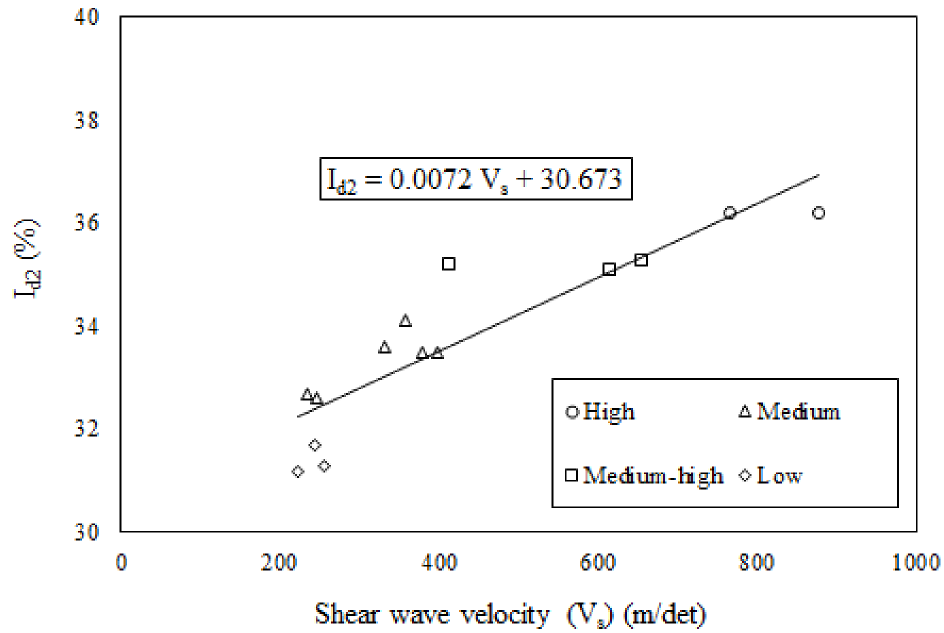

Figure 33. Relation between shear wave velocity (V_s) and slake durability index (I_{d2}).

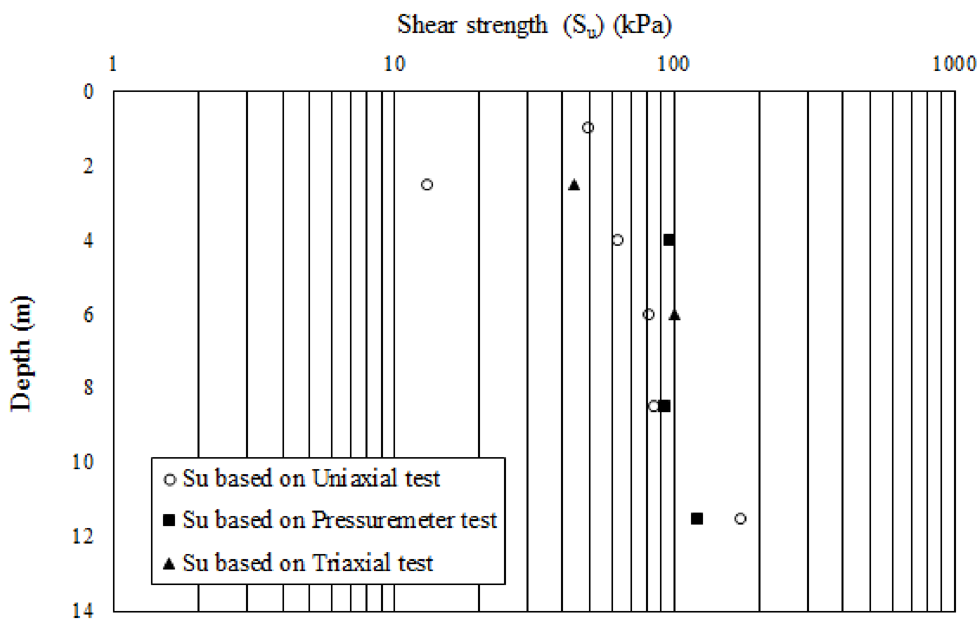

Figure 34. Comparison of undrained shear strength (S_u) between uniaxial; pressuremeter; and triaxial tests.

range of 1.65 to 2.5.

Coefficient of permeability (k)

Several studies (Brace, 1978; Davis and De Wiest, 1966; Serafim, 1968; Waltham, 1994) show that clayshale in natural condition using falling head test in laboratory and *in situ* test, is an impermeable. The k value is varied in order of 10^{-9} to 10^{-15} m/s. Test results show that the effect of cementation would lead to an increase with clay content and cause a decrease in permeability value

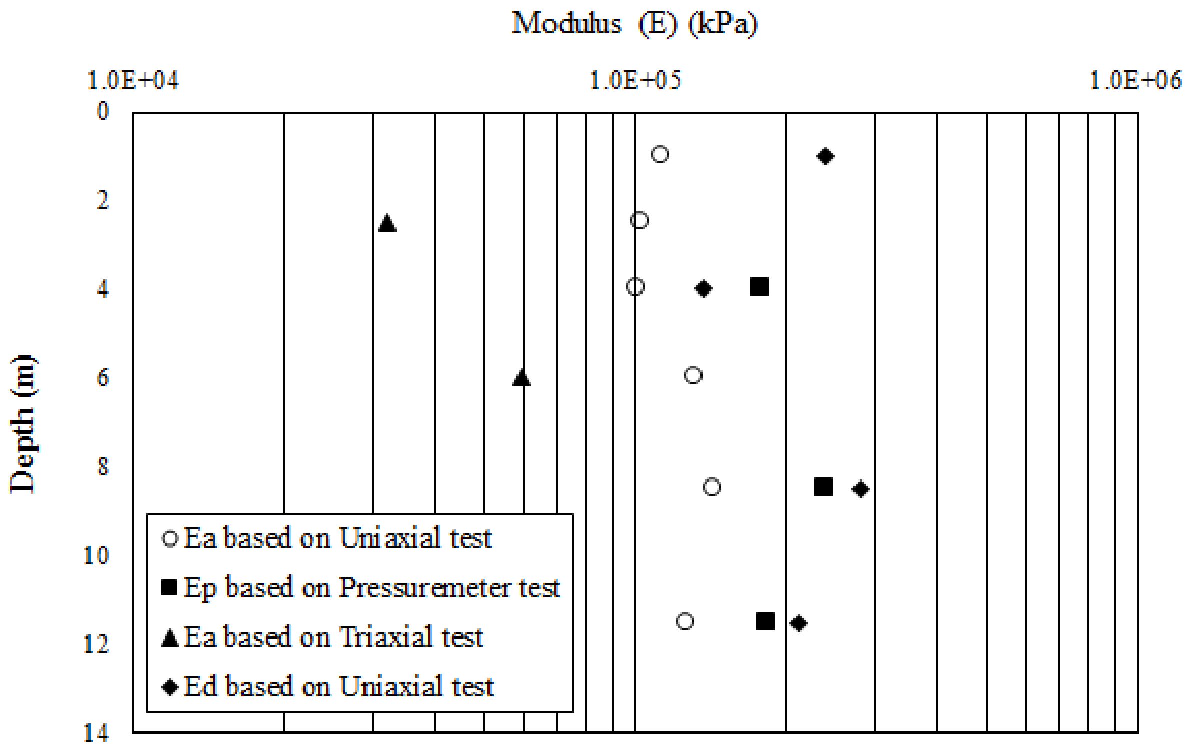

Figure 35. Comparison of modulus (E) between field and laboratory tests.

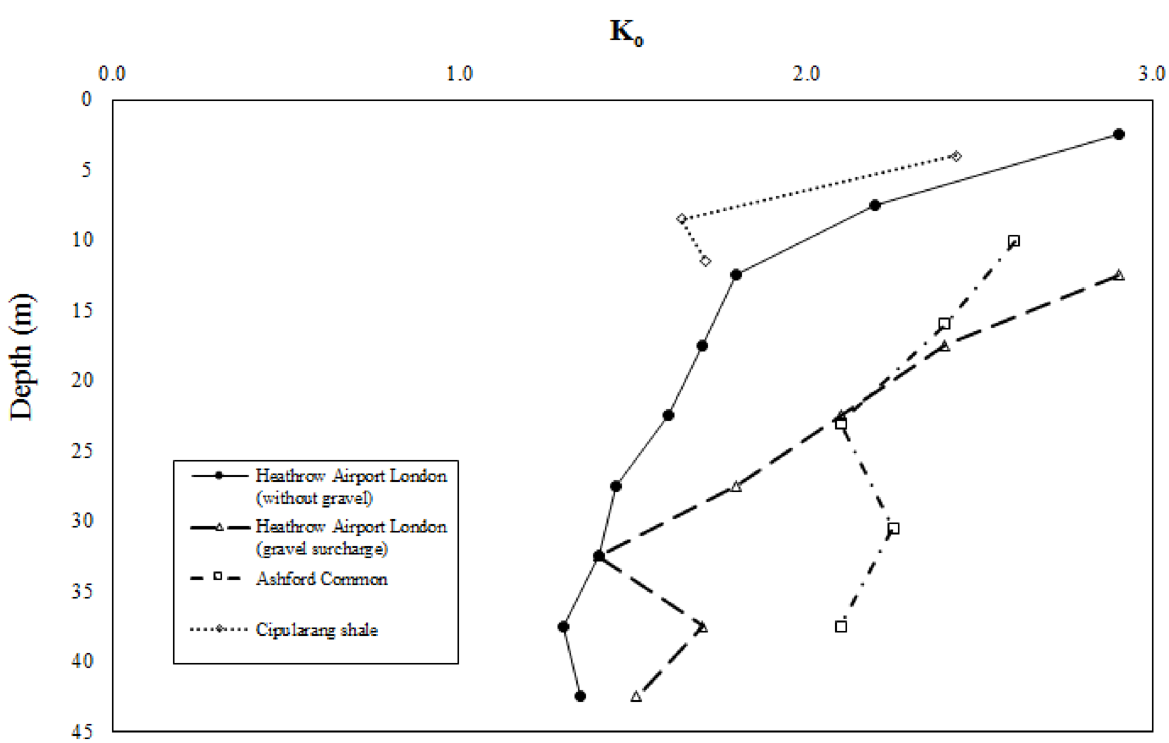

Figure 36. K_o profiles for the London Clay at Heathrow Airport London and Ashford Common (Hight et al., 2003) compared with Cipularang clayshale.

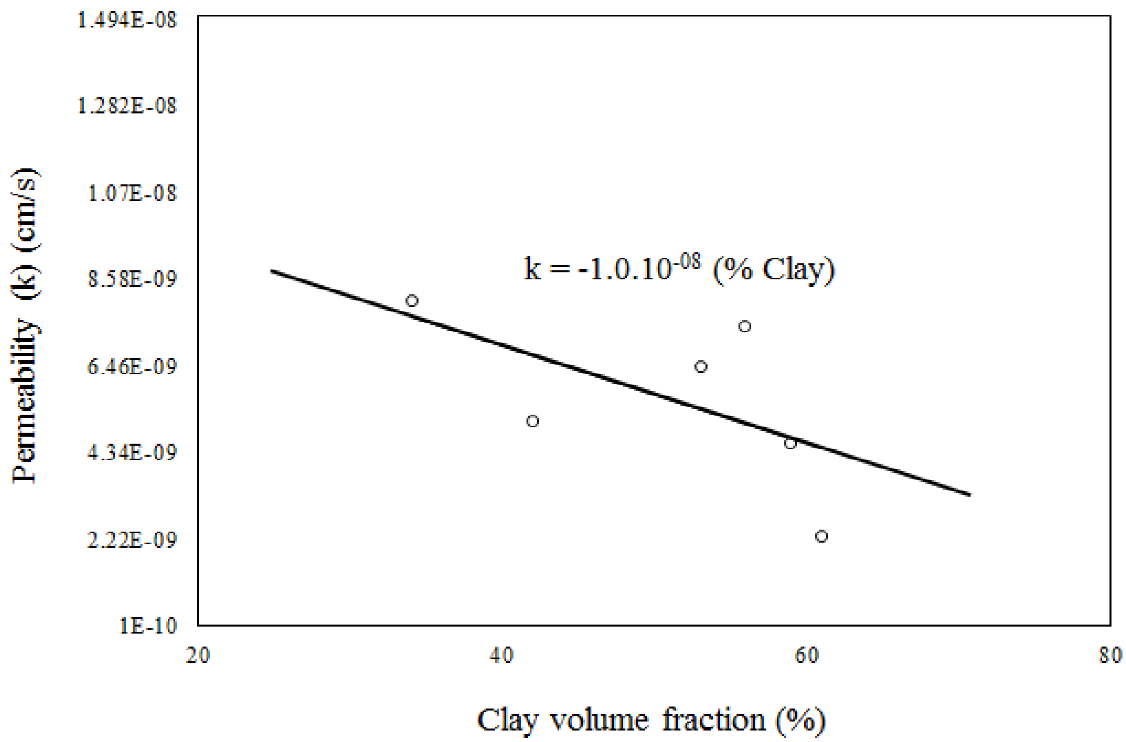

Figure 37. Permeability data versus clay volume fraction.

(Figure 37).

Consideration of slope stability

Using the laboratory and field tests mentioned earlier, several researchers (Knights et al., 1977; Laguros and Komar, 1980; Franklin, 1981) have proposed procedures for slope design on degradable materials. Although their proposal does not have the benefit of a wealth of engineering precedent and experience, they provide general guidelines.

Based on Figure 38, Franklin (1981) suggested that the maximum recommended slope of Cipularang shale is around 2H : 1V and 3H : 1V , or 25 to 35° according to shale rating values. Patterns field collapse is a landslide of blocks, translational, and circles. According to their observations, generally the field landslide lies at the boundary between the decayed shale and unweathered shale. Little or no stability problems were found where slab or block slaking dominated (degradation to thick, blocky fragments). Where chip slaking was dominant (degradation to thin, flat segments), the mass appeared to be relatively stable. The chips form an interlocking matrix which is resistant to bulk movement. When slaking to inherent grain size(degradation to fine-grained particles) was found to be the primary mode, stability problems were observed, as evidenced by slips, slides, and similar features (Perry and Andrews, 1982, 27). Hopkins (1988) found that the natural water content of an unwethered shale was a good predictor of important engineering properties. The behavior of these complex materials is experienced and enginering judgement for interpretation of the results.

Some recommendations for the design parameters of slope stability analysis are indicated in Table 3.

Some considerations required to avoid landslide in study area are to prevent the rain water absorbed by soil when excavation works are performed at clayshale area. One of the methods is preparation of a surface drainage system.

All excavation works tried do not exceed clayshale layers. It is suggested to use blanket layer or geotextile to avoid clayshale layers which could be exposed to air and water which could lead to slaking.

DISCUSSION

Based on several studies on clayshale behavior (Taylor, 1948; Terzaghi, 1967; Piteau and Peckover, 1978; Franklin, 1981; Bates and Jackson, 1983; Leet, 1971; Soewartojo et al., 1973; Deen, 1981; Walkingshaw and Shanti, 1996), geological classification (Mead, 1936;

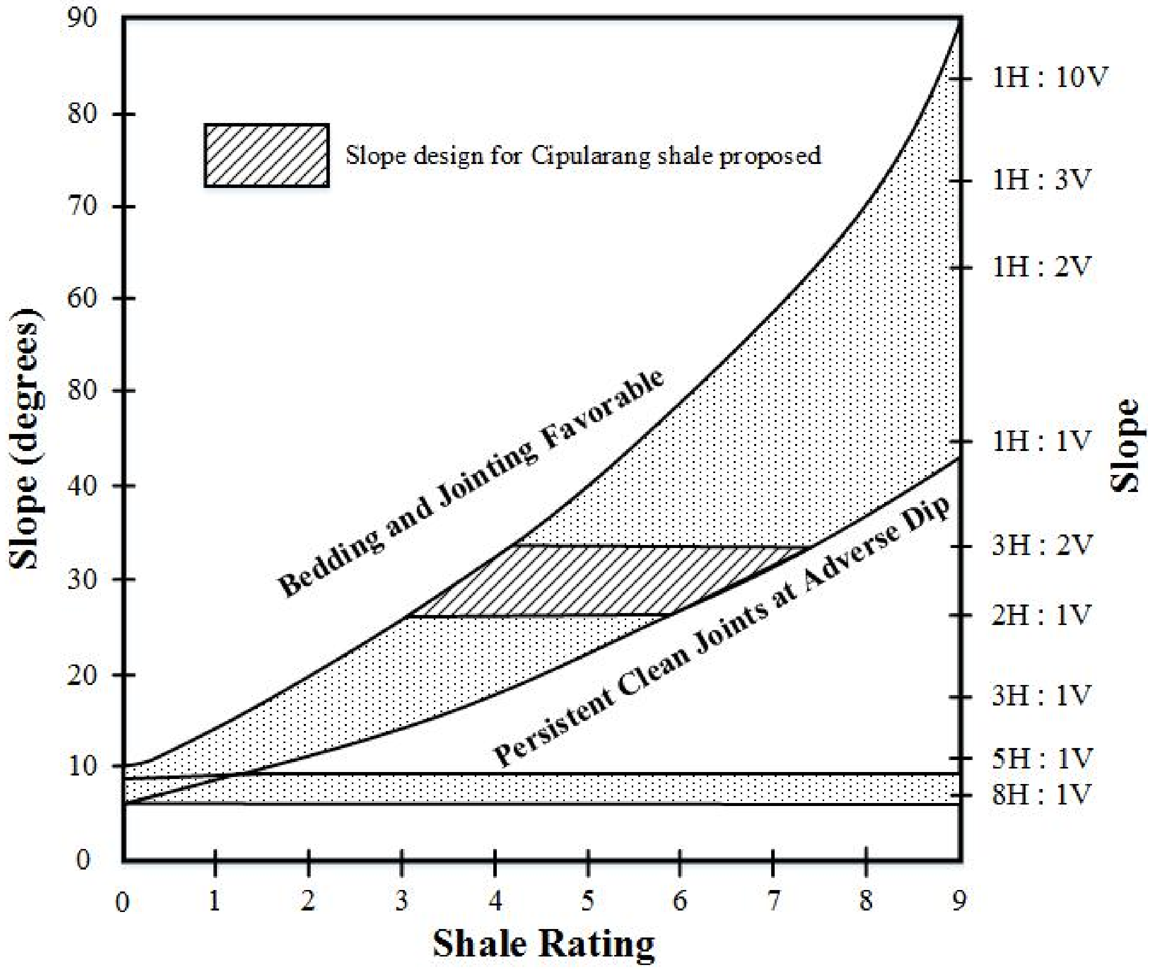

Figure 38. Stable zones of cut-slope as function of character of Cipularang shale (Franklin, 1981).

Table 3. Design parameters.

No.	c' (kPa)	ϕ (^{o})	c_r' (kPa)	ϕ_r' (^{o})	Ref.
1	15-20.0	18-21	0-0.1	17.5	Gartung (1986)
2	7.5-10.0	21-23	1.0-2.0	13-16	Stark and Duncan (1991)
3	3.5-7.0	15.5-20	0-0.1	10-12	Skempton (1977)
4	22-25	20-22	1.0-1.2	11-12	Peterson (1954)

Ingram, 1953; Philbrick, 1950; Krumbein and Sloss, 1963; Folk, 1968; Underwood, 1967; Belviso et al., 1977; Wentworth, 1922), and geotechnical classification (Twenhofel, 1939; Terzaghi, 1936, 1946; Muller, 1964; Bjerrum, 1967; BSI, 1957; Gamble, 1971; Deo, 1972; Morgenstern and Eigenbrod, 1974; Shamburger et al., 1975; Deen, 1981; Botts, 1986), material in Cipularang is categorized as soil-like *clayshale* with durability varying between low and high for unweathered samples. There is a tendency for the slake durability to be higher in the deeper zone (Keller, 1976; Peck et al., 1974; Johnson, 1969; Bjerrum, 1967, US Army, 1956, 1990; Vargas, 1953; Hendron et al., 1970). Weathering process significantly reduces durability of clayshale (Einsele and Wallrauch, 1964; Attewell and Farmer, 1976). The reduction can reach up to 50%. According to Gamble classification, Cipularang clayshale is categorized as a clayshale with a very low durability (I_{d2} < 30%). Based on the strength characteristic, Cipularang clayshale can be classified as very soft rock with low rigidity (Deere and Miller, 1966). In terms of geology, Cipularang clayshale is easily a weathered rock (Franklin and Chandra, 1972; Deo, 1972; Moriwaki, 1975) using ASTM D 4644 – 04.

Durability (I_{d2}) of rock data is lower than 30%; it was difficult to perform an uniaxial compression test because sample is fragile and collapse in test preparation, so that the uniaxial test resulted data cannot be obtained. For point load test, the information of $I_{s(50)}$ also cannot be obtained because the sample was directly damaged when the test was performed. Slaking effect may influence the durability and strength of clayshale. Increasing moisture content is very influential to reduce durability w shown by increasing pore volume and decreasing of strength clayshale.

Permeability (k) of Cipularang clayshale produces result ranging between 10^{-9} and 10^{-15} m/s using the falling head test. These studies are almost the same with several studies (Brace, 1978; Davis and De Wiest, 1966; Serafim, 1968; Waltham, 1994). Test results show that the effect of cementation would lead to an increase with clay content, and cause a decrease in permeability value.

Based on hardness and durability value, Cipularang clayshale may classified into 2 (two) types, such as highly weathered rock and slightly weathered rock between low to high. Increasing of V_s value shows the enhancement of durability (I_{d2}) value.

Coefficient of pressure at rest (K_o) at clayshale Cipularang shows that the value is tendency to decrease with depth. K_o value varies between 1.6 and 2.5 which is almost equal to London Clay (Skempton, 1961). Horizontal stress for this case is greater than the vertical stress.

Axial modulus from uniaxial testing is smaller around 1/7 times than axial modulus from laboratory ultrasonic test. These results are close to that of Deere et al. (1967) studies. Whereas, comparison between pressuremeter modulus (E_p) and axial modulus (E_a) from uniaxial test is 1.4 to 1.9. All values depend on mechanism of stress-strain during testing and test method to determine modulus values.

CONFLICT OF INTERESTS

The authors have not declared any conflict of interests.

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Full Length Research Paper

Enhancing benefits from biomass wastes within small-medium scale coffee processing factories in Kiambu County, Kenya

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Coffee processing is associated with various sustainability challenges largely due to high water and energy demand, biomass waste generation and lately low returns to farmers. The number of farmers actively involved in coffee production in Kenya is declining. Despite this trend, this paper reports on the potential of maximising benefits to coffee farmers by using coffee processing wastes (pulp and husks) in energy and agricultural services. A survey research design targeting six small to medium scale factories selected based on common criteria and 252 respondents was used. Results showed that about 210 tons of unprocessed coffee was received cumulatively in all the six factories surveyed per year. Processing generated approximately 51% biomass waste from the total input. Direct disposal of this waste to land contributes to direct environmental pollution. Accumulation of coffee husks and pulp was attributed to low awareness of the various uses these wastes have been successfully put elsewhere in the world. Opportunities for increasing benefits to farmers and reduced environmental loads exist in the conversion of coffee husks into branded briquettes for domestic energy supply, and pulp into fortified organic fertilizer for increased land productivity. Kiambu County government needs to invest in these two options through technological innovations and commodity specific extension service that is aligned to global sustainable production and consumption patterns.

Key words: Coffee processing biomass wastes, sustainability benefits.

INTRODUCTION

Coffee production in Kenya has been on the decline since 2009, a trend attributed to among others factors erratic weather, conversion of coffee land to real estates and high costs of inputs (Republic of Kenya, 2013a). However, estimates from the International Coffee Organization (2010) indicate that the total production of coffee from exporting countries increased in years 2000 and 2010 from 112, 991,000 and 133,065,000 bags,

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respectively with Kenya exporting 51,000 metric tons of coffee beans in 2010 alone. In Kenya, more production is being witnessed in smallholder systems as large plantations, especially those in proximity to the city Nairobi, give way to real estate development. Despite this trend, projections indicate that Kenya's coffee production in 2016/2017 will increase to 700,000 bags up from 600,000 bags in 2015/2016 (USDA-FAS, 2016). Fluctuations in global market prices have nevertheless often been detrimental to small-scale farmers. In addition, organic waste generated from coffee processing and other crop specific postharvest processes are challenges that need to be addressed in-line with environmental sustainability concerns (Republic of Kenya, 2013b), hence the gist of this paper.

The main sustainability concerns in coffee processing are as a result of intensive use of pesticides and poor disposal of waste products. About 99% of the biomass waste produced, mainly untreated pulp and husks is discarded on land. Further coffee processing consumes high quantities of water and energy. According to Shitanda (2006), over 200,000 tonnes of pulp at 77% moisture content and 2,300,000 litres of polluted water are released into the environment everyday in Kenya. This is equivalent to pollution caused by 1.2 million people per day. On average 45.5 kg of green coffee requires between 1000-2000 L of water, 12.5 kWh of electricity and 0.07 cum of firewood for processing (Instituto del Cafı de Costa Rica -ICAFE, 2006). A survey of rivers between Nairobi and Thika towns in Kenya showed that they were all polluted with coffee waste with Biological Oxygen Demand (BOD) levels of more than 100 mg/l. The unpolluted rivers had BOD of 4 mg/l (Wrigley, 1988). Although a river of 10 mg/l is considered significantly polluted, the maximum allowable limit of effluent discharge into the environment is 30 mg/l (BOD 5days at 20°C) according to Kenya's National Environment Management Authority (NEMA) Standards (Republic of Kenya, 2006). In pursuit of sustainable goals 6 and 12 (https://sustainabledevelopment.un.org) such large volume of water can be treated and reclaimed for other uses particularly in a water scarcity nation like Kenya (http://www.unep.org/dewa/vitalwater/article83.html), this waste water can be treated and reclaimed for other uses. Establishing facilities capable of improving the overall efficiency of coffee processing with focus on waste reduction is costly and may not be affordable to majority small-scale farmers. Opportunities for bulking raw material lies with factory level operations. Studies elsewhere indicate that coffee husk and pulp can be used as organic fertilizer, domestic fuel, and for biogas generation (Ulloa et al., 2003; Pandey et al., 2000; Kaliyan, 2009, Sanchez et al., 1999; Sorby, 2002), thus reducing the burden of waste disposal.

Although coffee processing has significant sustainability challenges, major coffee producing nations such as Brazil have made significant efforts to treat coffee waste water and utilize solid biomass waste, thus contributing to reduced environmental pollution and burden on virgin resources (Cofie et al., 2005; Padmapriya et al., 2013). Further, since the middle of this century, efforts have been made to develop methods for coffee waste treatment and management, and also its utilization as a raw material for the production of vinegar, biogas, caffeine, pectin, peptic enzyme, protein, compost and feed for producing polysaccharides and mono-saccharide. While multiple benefits from using and adding value to agricultural wastes are appreciated in densely populated nations like India (Sindhu and Shehrawat, 2015), a key adoption limitation in most developing countries is lack of awareness and lack of detailed regulations on crop specific agricultural wastes management for environmental protection (Khanh and Thanh, 2010). Kenya lags behind in this regard, which translates into missed opportunities towards increased environmental and socio-economic benefits to farmers. Availability of this knowledge implies that new extension approaches that address the entire crop value chains with farmers owning key aspects thereof like is the case in soil improvement demonstration plots by the Alliance for a Green Revolution in Africa (AGRA) and International Institute of Rural Reconstruction (IIRR) (2014) may benefit coffee systems. Milling sites can be converted into extension service demonstration sites (plots) for maximized benefits from coffee wastes. For instance, the Gusii Coffee Farmers Co-operative Union is among progressive millers in Kenya who have embarked on processing of coffee husks into branded charcoal briquettes (Gusii Coffee Charcoal Briquettes - 100% Organic) as part of its efforts to protect the environment from the menace of dumping husks at the Union's coffee milling site (Oroko, 2015). Here-in is an initiative that the Ministry of Agriculture and the National Environment Management Authority (NEMA) could isolate for upgrading into a demonstration centre in order to scale-out and scale-up environmental and economic benefits from coffee wastes across all millers and supply farmers in the country.

Already this prospect of energy from coffee wastes has attracted entrepreneurs dealing with different products along the coffee value-chain, such as production of briquetting machines improved cookstoves for using such briquettes and product distribution and consultancy services (Youth Agro-environmental Initiative, 2015). Although the by-products of coffee processing include mucilage, which is part of waste water, the focus of this study was the potential of using pulp and husks in agricultural and energy services at the farm level. Suffice is to indicate that mucilage which is removed through the fermentation process makes waste water have serious environmental problems due to the high acidity generated

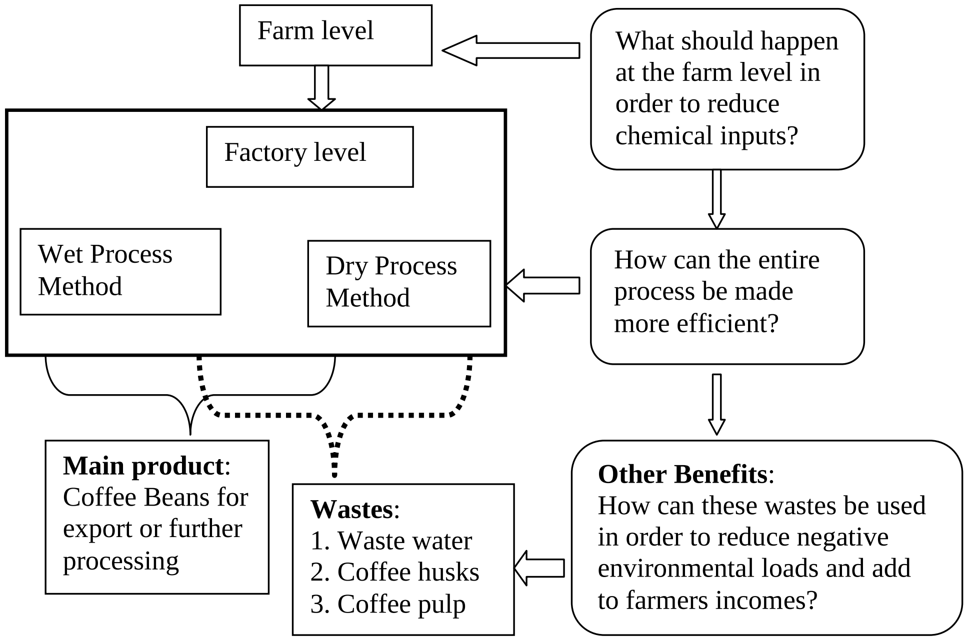

Figure 1. A life cycle management framework in coffee processing.

and also Biological Oxygen Demand (BOG) and chemical oxygen demand (COD) (http://www.coffeeresearch.org/agriculture/processing.htm; http://www.saiplatform.org/uploads/Library/6Coffee_Waste_Water_tretamentV4.pdf). For this paper, the focus was how to maximise benefits from coffee husks and pulp in the context of sustainability thinking.

METHODOLOGY

This study was done in Kiambu County, located within Central Kenya highlands. Its socio-economic activities are greatly influenced by its dense population, rich agricultural potential and proximity to Nairobi, the capital of Kenya. This County was selected because of its fame as a coffee growing and processing region, and increasing popularity of commercial peri-urban agriculture, which could tap into coffee biomass wastes as unique inputs. Since the main goal of this study was to assess general trends in the flow of pulp and husks, a survey design was adopted. Data was collected using interviews, questionnaires and environmental check lists. Primary data was obtained from a random sample of 252 respondents including 240 farmers and 12 factory operators obtained from the six study factories, namely *Karia, Gititu, Riabai, Gatei, Ndarugo and Ndumberi*. These factories were selected for being in the category of small to medium processing status (that is, capable of handling 500-3000 tons of coffee per year), and for having been operational with accurate records since 2008.

Quantities of coffee husk and pulp in each factory were calculated as percentages of the total amount produced per year. Variance at factory level with respect to the amounts of coffee solid waste and demand was also determined across the factories. The research plan used is summarised in Figure 1. Observation checklists were used to document status of coffee processing infrastructure, various uses of coffee husks in the factories and the surrounding area, and critical environmental changes associated with coffee processing. Data was analyzed mainly by use of descriptive statistics since the main goal was to determine general trends in the key variables under investigation.

RESULTS AND DISCUSSION

Sustainability concerns

At the farm level, the study revealed that instability of coffee prices leads to low income generation from coffee growing. This translates into poor investment in farm inputs such as agro-chemicals and protective gear, as attested to by about 68% of the farmers interviewed (Table 1). On farm occupational health and safety standards remain items of concern. Since coffee farming requires high chemical input to regulate pests and diseases, farmers are at high risks of serious health concerns from exposure to toxic substances.

High demand for water in coffee processing is not a new challenge. The concern among millers in Kiambu County is the lack financial capacity for technological solutions to decrease and or treat the volume of waste water as well as turn solid biomass waste into other products such as fertilizer. Overall coffee farmers viewed sustainability concerns from a financial and economic perspective only, when negative environmental effects are likely to be most significant. In terms of marketing,

Table 1. Sustainability concerns for coffee processing life cycle.

Life cycle stage and activities	Sustainability Concerns (Economic, Social and Ecological)
Farm level	- Expensive agro-chemicals - Health risks due to minimum use of protective gear - Soil pollution due to copper-based agrochemicals - Water pollution from agro-chemical carried in sediment
Factory level	- Inadequate support infrastructure - Health and safety standards of coffee workers - High energy and hence threat to tree cover - High water demand amidst other competing uses - Odour from accumulating coffee pulp - Water pollution from disposed waste water
Marketing	- Declining and volatile coffee prices
Waste management	- Lack of a robust management framework - Inadequate protective gear - Poor disposal of waste water, pulp and husk - Negative environmental load particularly due to accumulating pulp

global fluctuations in prices and existence of multiple brokers in the marketing chain translates into diminished net returns to farmers.

Inventory analysis indicated that more than 8,000 tons of husks and pulp are generated every year in Kiambu County from using both wet and dry processing methods at factory level. All factories processed coffee using the wet method except *Ndarugo* and *Riabai*. As a result the amount of coffee husks generated in the two factories was significantly higher than other factories (Table 2). The husk generated in *Ndumberi, Gititu, Karia* and *Gatei* accounts for approximately half of the unprocessed coffee received. *Ndarugo* and *Riabai* husk generation is higher accounting for 57% of unprocessed coffee beans. On average the amount of husk and pulp generated in all factories in 2010 was 122.76 tons. With such availability of raw material, benefits along the coffee processing value-chain can be extended.

All the six factories recorded high wastage rates producing very little fine coffee in comparison to the unprocessed coffee received. Factory records showed that more than 8,000 tons of husks and pulp are generated every year in the County from using both wet and dry processing methods. The amount of coffee husks generated at *Ndarugo* and *Riabai* factories was significantly higher than other factories because the two used dry processing method. All factories except Gatei registered a general decline in raw coffee production from 2008-2010. This is indicative of changing land use in peri-urban areas in Kenya in favour of real estates. Overall about 56% of husk and pulp was generated, which translated into avoidable environmental burden were mechanisms of turning such waste into useful products available.

Relative importance and uses of coffee husks and pulp in Kiambu County

The utilization of coffee husk generated varied from one factory to another. Only 3% of the solid biomass was directly used as biomass fuel within the factories. About 13% of solid biomass waste was sold to external users, who used it in poultry farming. Up to 40% of the pulp and husk was disposed to the land, signifying lack of awareness on extra value or recycling technology for these resources (Figure 2). In addition to direct disposal of waste on land, limited knowledge on other ways of composting could account for the widespread use of open-pile approach, which reduced the pulp and husks to low grade organic fertilizer. Comparative assessment showed that more husk and pulp were used in agricultural services than in energy services (Table 3).

This is attributed to the high cost implications when it comes to biogas production and briquetting technologies. In addition the practice of using agricultural residues for manure and compost has been practised for a long time in Kiambu and thus become an acceptable culture unlike the use of agricultural residues for energy production at factory and household levels. This is indicative of the need for innovation and capacity building towards increasing benefits of coffee waste in energy service provision and other possibilities along coffee value chain. Compared to factory level, more solid biomass was used for composting as reported by about 47% of the respondents (Figure 3).

Table 2. Factory records of quantities of raw coffee and generated husks and pulp (tons).

Name of Factory	Year	Raw coffee received (tons)	Processed coffee (Tons)	Husk/Pulp generated (tons)	Husk/Pulp as % of raw coffee
Ndumberi	2010	94.70	24.00	36.75	38.80
	2009	100.13	48.60	56.07	56.00
	2008	102.90	43.20	57.62	56.00
Gititu	2010	45.00	18.50	25.20	56.00
	2009	51.70	21.70	28.99	56.10
	2008	53.00	20.50	29.70	56.00
Karia	2010	19.00	7.90	10.64	56.00
	2009	22.00	10.10	12.32	56.00
	2008	--	--	--	--
Ndarugo	2010	19.60	7.90	10.97	55.95
	2009	24.30	11.30	13.61	56.01
	2008	22.80	9.40	12.76	55.96
Riabai	2010	15.00	7.30	8.40	56.00
	2009	19.00	9.40	10.64	56.00
	2008	21.50	10.10	12.04	56.00
Gatei	2010	36.00	18.60	20.16	56.00
	2009	27.50	13.80	15.40	56.00

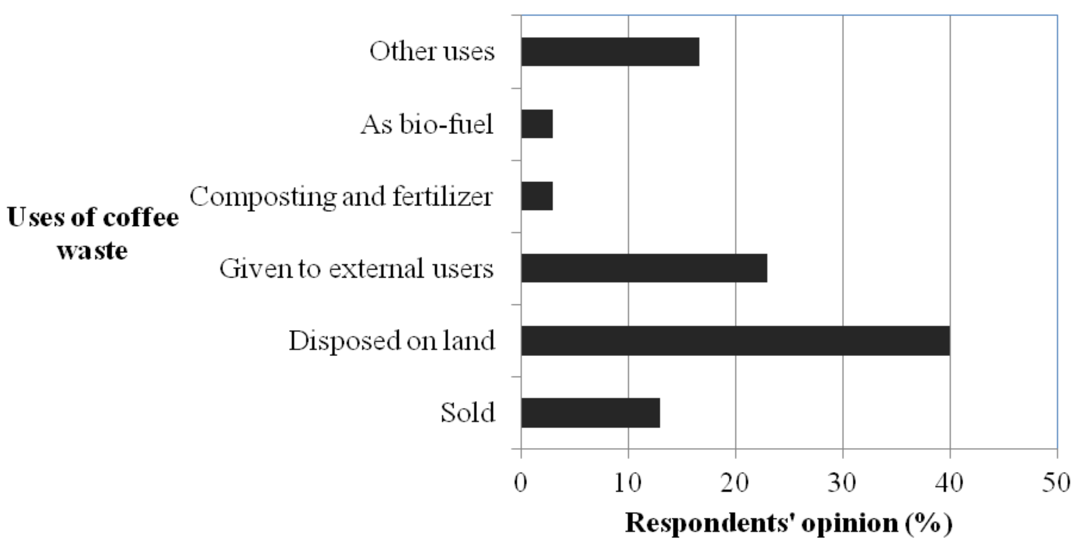

Figure 2. Relative importance of the uses of coffee husks and pulp at factory level.

Ndumberi Youth Group was the only respondents' category using coffee pulp for briquette production, accounting for only 0.3% of the respondents. This statistics is expected given the industrial nature of briquette production and commensurate skills required which ordinary farmers do not have. Lack of knowledge of other forms of composting technologies could account for the widespread use of open pile system approach.

Table 3. Comparative use of coffee husk/pulp in agriculture and energy services (%).

Agricultural Services		Energy Services	
a. Organic fertiliser	27.10	a. Direct Biomass	13.70
b. Animal Feed	5.50	b. Biogas	4.50
c. Direct disposal on land	33.10	c. Briquettes	12.20
Total	**65.70**	**Total**	**30.40**

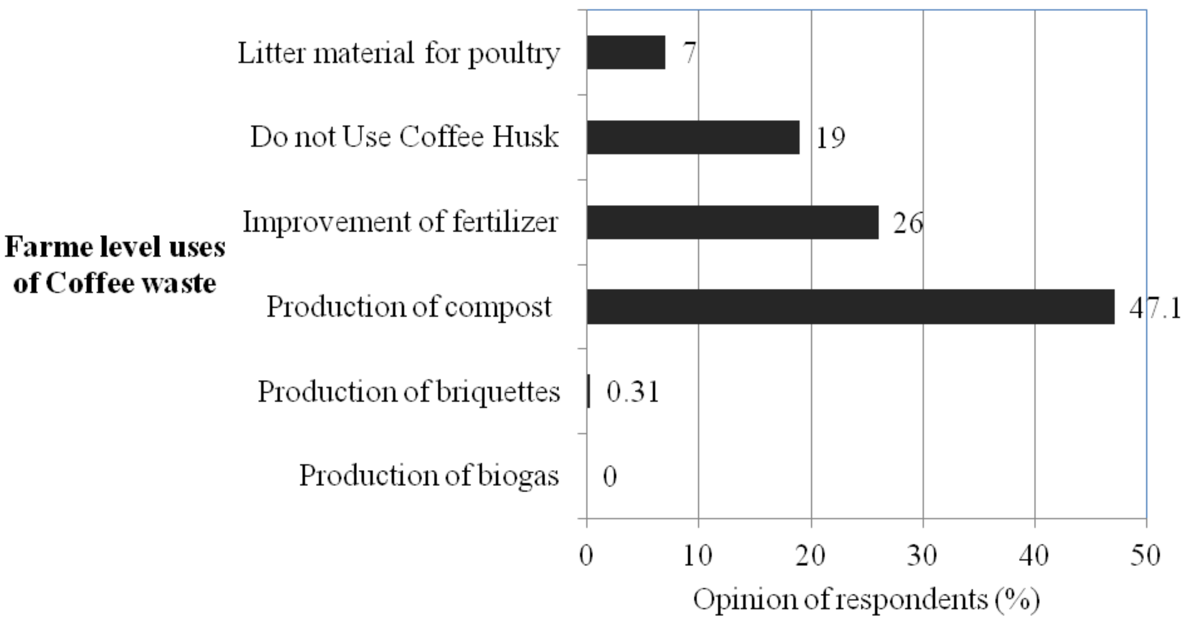

Figure 3. Comparative use of coffee husk/pulp among farming households and business users.

Increasing awareness and enhancing capacity on available technologies for using biomass waste for energy and agricultural services can contribute to husk and pulp management at household level.

Unlike Kenya, studies conducted in Costa Rica and Vietnam show that coffee pulp and husk are used in many more applications such as biogas production, mushroom growing, mulch, and animal feed among other uses (Pandey et al., 2000). With such readily available raw material, Kenya is missing out on other eco-based business opportunities with potential to impact farmers and their environment positively. This calls research and capacity building around multiple uses of coffee wastes along the value chain.

Challenges of using coffee husks and pulp in agricultural ecosystem

The outstanding challenges facing the use of coffee husk and pulp in the six factories were limited financial resources for investment in waste management, lack of requisite equipment in material management, low demand of husks, and lack of trained personnel in overall agricultural waste management (Figure 4). Use of pulp and husks in energy services provision was constrained by lack technical knowledge and availability of other sources of cooking energy like kerosene, Liquefied Petroleum Gas (LPG) and wood fuel. Faced with challenges of climate change and declining availability of wood fuel, investing in biogas systems is one approach where the potential of pulp and husk could yield more economic and environmental benefits.

In Vietnam, biogas from pulp and parchment generation is used to produce both electricity and heat (Ali, 2004). However, respondents indicated that pulp and husks from coffee processing cannot be compared to other solid waste materials such as metals, paper, and some plastics, which have high demand from recycling traders within Kiambu. Further, coffee husks and pulp are also bulky and thus cumbersome and difficult during handling, transportation and storage. Respondents also

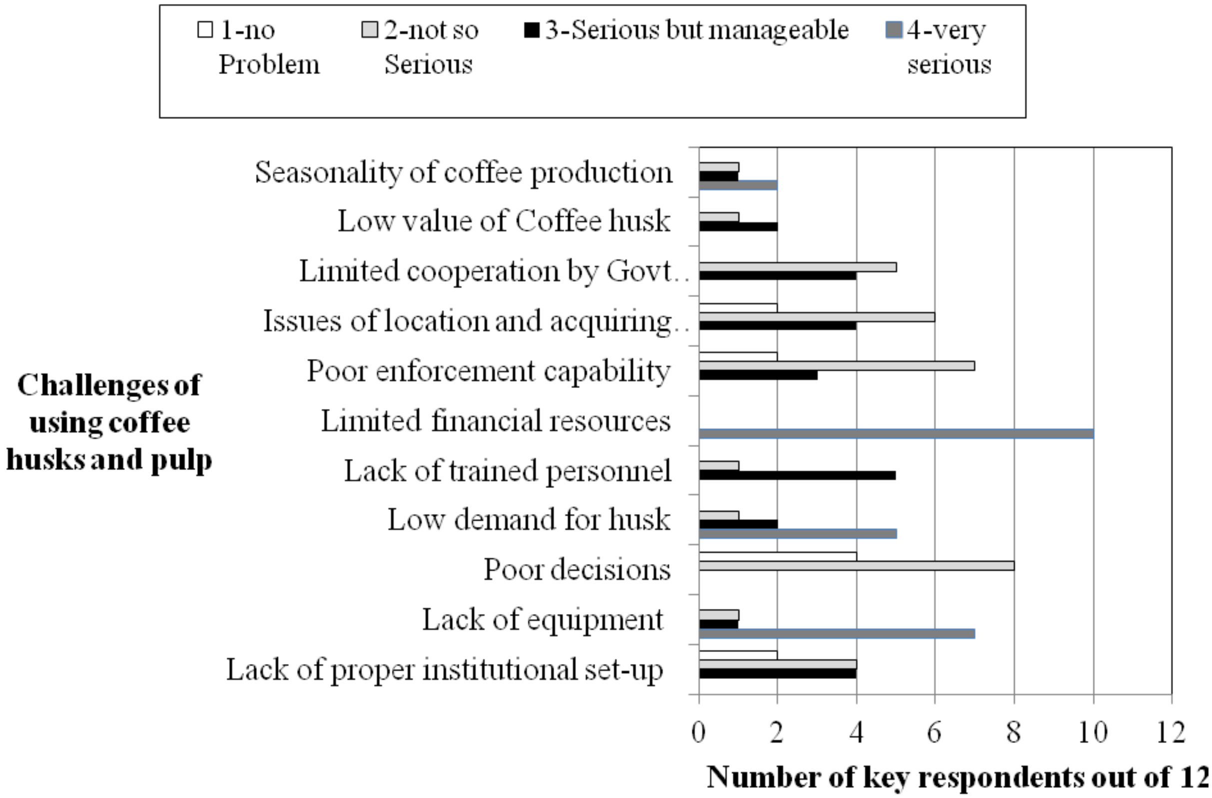

Figure 4. Factory operators' opinions on the challenges of using coffee husk and pulp.

blamed the low use of coffee husks and pulp in agriculture to risk of contamination from caffeine and tannins. More than 80% of respondents were also not aware of companies that use waste or persons who could collect the waste, hence the apparent accumulation of this waste. More than 60% of the respondents were not aware of biomass alternative devices and did not know what happened to collected wastes. About 55% did not think that coffee wastes could be a source of good income, but interestingly, more than 70% of the respondents were willing to pay for waste management services (Figure 5). This opinion could be informed by their perhaps higher understanding and concern for environmental quality and human health.

It seems that with good capacity and competence building, farmers and factories could invest in recycling and or re-use of coffee wastes as sources of extra income and in the interest of environmental health. The coffee factories relied on limited financial resources and therefore lacked the ability to manage the amount of coffee husk and pulp produced. On average 45.5 kg of green coffee requires between 1,000-2,000 L of water, 12.5 kWh of electricity and 0.07 cu. m of firewood for processing (Instituto del Cafı de Costa Rica - ICAFE, 2006). This is exacerbated by lack of equipment and infrastructure in the factories. Incorporating waste management as an integral part in coffee processing can establish innovative and feasible approaches which have potential to maximize use of pulp and husk in Kiambu. Mobilizing resources for biogas development at selected factories has merit in the context of clean production mechanisms as envisaged in sustainable development goals.

In terms of the use of coffee solid wastes, the survey revealed that planning and economic factors were the most limiting constraints and in particular lack of awareness about alternative uses of wastes and lack of modern equipment in waste management (Figure 6).

About 20% of farming households raised concerns about using urban composts for fear of contamination and hence risk to consumers of produced crops. In addition, about 70% of the farmers had a positive attitude towards compost but were not willing to pay for the product as opposed to chemical fertilizers. Further 87% of respondents were not aware of alternative energy devices for domestic cooking and heating that is available, despite many agreeing that biomass solid coffee waste can be profitably utilized. Combined efforts by the public extension, environmental agencies, research and training organisation is needed to raise awareness in

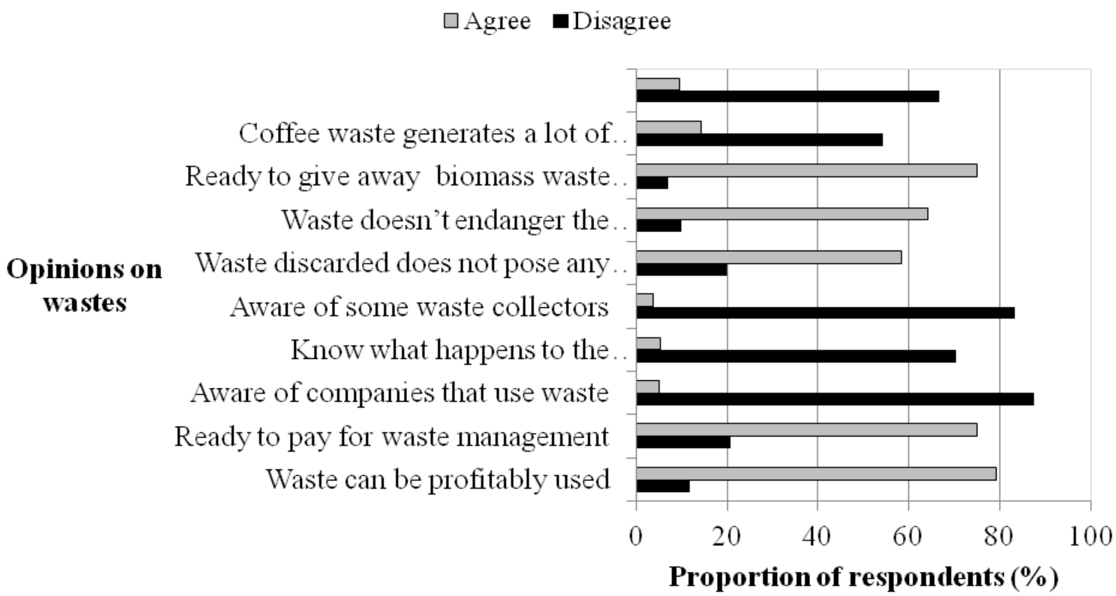

Figure 5. Respondents' opinions on various aspects of biomass coffee waste (%).

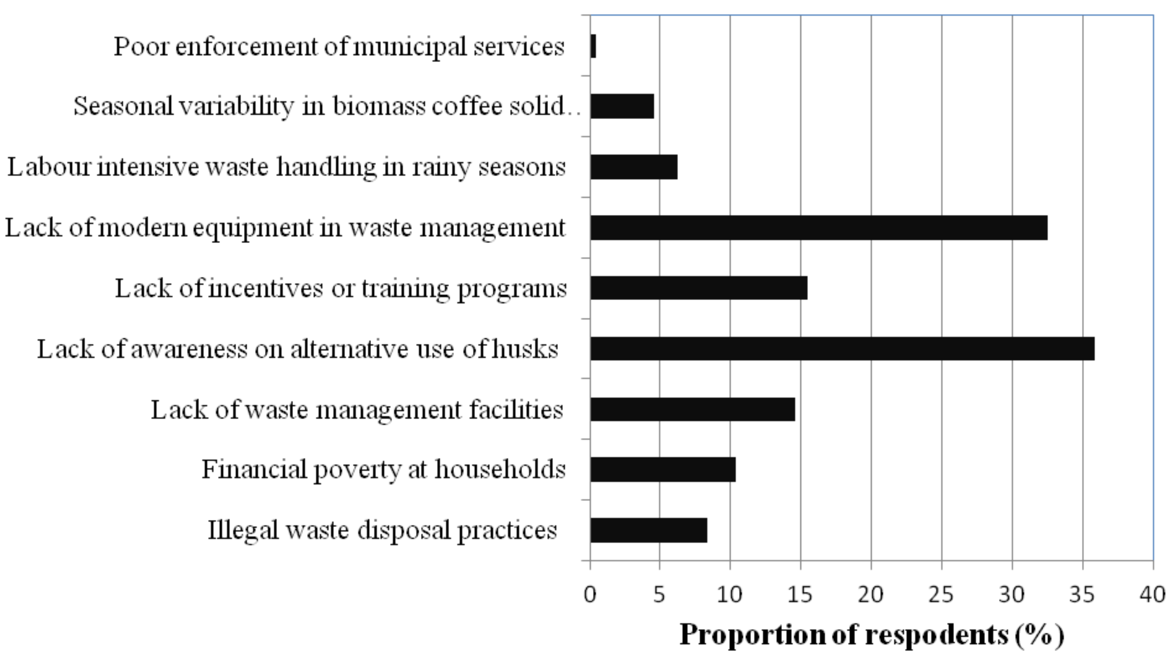

Figure 6. Factors influencing use of coffee solid wastes.

the potential of coffee wastes in environmental enhancement and income generation.

CONCLUSIONS AND RECOMMENDATIONS

Solid biomass wastes from coffee processing constituted about 51% of the total raw input. About 40% of this waste is routinely disposed directly to the land hence contributing to avoidable pollution. Success stories elsewhere indicate that with proper planning and investment, these amounts of coffee wastes can be used in particular compost, briquettes and biogas at selected factories, being inherent bulking centres. Accordingly some factories can be strategically selected, re-organised and re-designed to include in-situ compost and biogas production systems. While compost can be sold to farmers or distributed to them based on corporate social responsibility principles, the biogas can be distributed to homesteads in proximity to the design factories to ease their reliance on wood fuel.

For not being bulky, coffee husks can also be used for mass production of branded briquettes in-situ or elsewhere and marketed as an alternative source of clean domestic energy. The contribution to global effort against reliance on wood fuel, increasing tree cover and mitigation of climate change effects cannot be overemphasised. Similarly, waste water that is conventionally discharged into water bodies can be sufficiently treated and used for irrigation in crop farming. All these under-utilised agri-business innovations call for commodity specific extension service, which should be deliberately aligned to sustainable development goals focussing on sustainable production and consumption agenda.

CONFLICT OF INTERESTS

The authors have not declared any conflict of interests.

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Full Length Research Paper

The effect of *Amaranthus hybridus* on fluoride removal by iron (III) salts as fluoride coagulants

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The use of iron (Fe) (III) salts as fluoride coagulants in water is challenged by the requirement of high pH for maximum efficiency. At their natural pH, these salts have low fluoride removal efficiency. This study examines the effect of amaranth plants on enhancement of the defluoridation efficiency of Fe (III) salts as coagulants. *Amaranthus hybridus* plants were suspended in fluoride water treated with varying concentrations of Fe (III) with its roots immersed completely in fluoride water for varying time from 720 to 1440 min. The study shows that fluoride coagulation by Fe (III) in the absence of plants is limited to 10%, whereas when plants were introduced, it increased from 10 to 40%. These results suggest that amaranth plants enhance the defluoridation efficiency of Fe (III). This enhanced removal may be attributed to increased coagulation effected by exudates released by plant root which contain organic compounds and CO_2 or charged root surfaces by the formation of Fe (III) oxide film. The exact factor that has a major contribution to enhanced removal observed remains to be subject of further studies.

Key words: Coagulation, defluoridation, iron (III) salts, phytoremediation, plant exudates.

INTRODUCTION

Fluoride occurrence in surface and ground water is a global problem and is a cause of fluorosis in both humans and animals (Fawell et al., 2006) with over 200 million people at risk (Fawell et al., 2006). Ingestion of fluoride concentrations greater than 1.5 mg/L is associated with health problems, the more notable being dental fluorosis (Murray, 1986). Even though fluoride rich food materials such as fresh vegetables, meat, milk and some of the cereals can contribute to the total ingestion of fluoride (Radha et al., 2010), it is drinking water which is identified as the main contributor to fluorosis occurrence in humans (McClure, 1936; Ruiz-Payan et al., 2005).

There are several options imaginable to evade fluorosis, namely, (i) using water sources with fluoride level less than 1.5 mg/L, (ii) dilution of fluoride water with fluoride free water and (iii) defluoridation of drinking water. Defluoridation of drinking water however, is the only practical option to overcome the problem of

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excessive fluoride in drinking water, where alternative water source is not available. The widely studied fluoride removal methods include coagulation by alum and lime (Fawell et al., 2006; Emamjomeh and Sivar, 2009; Behbahani et al., 2011), ion exchange/adsorption (Bhatnagar et al., 2011; Zhong et al., 2013) and membrane filtration (Alkan et al., 2008; Sehn, 2009). Other methods tested include phyto-remediation by aquatic plants such as *Hydrilla verticillata* under laboratory and field conditions (Sinha et al., 2000) and a combination of these methods. Defluoridation by phytoremediation method did not receive considerable attention from researchers, understandably due to low fluoride uptake by used plants reported (thence low defluoridation efficiency) (Sinha et al., 2000), although some plants in general are reported to have good fluoride bioaccumulation property (Njenga et al., 2005; Khadare and Rao, 2006; Yadav et al., 2012; Patil et al., 2014). Phytoremediation is well-known in municipal wastewater and heavy metals contaminated soils and water (Wuana and Okieimen, 2011). In these plants, introduction of chemicals that form complexes with destined contaminant has enhanced its removal (Nowack et al., 2006; Wuana and Okieimen, 2011).

On the other hand, water defluoridation by coagulation is widely applied in what is commonly known as the Nalgonda technique (Fawell et al., 2006). In this technique, a mixture of alum and lime are employed to flocculate/coagulate and precipitate fluoride from water (Fawell et al., 2006). Challenges associated with this technique include (i) alkalinity of the treated water and (ii) presence of fluoro-alumino complexes in the treated water. Fe (III), tricalcium phosphate and Moringa seed powder are among the studied fluoride coagulants (Boruff, 1934; He and Cao, 1996; dos Santos Bazanella et al., 2012). The use of Fe (III) in fluoride coagulation is not widely reported in literature even though it is a potent fluoride coagulant. Since the optimal conditions for Fe (III) coagulation is in alkaline media, the low Fe (III) fluoride coagulation efficiency reported by Boruff (1934) could be due to this factor. This alkaline media is achieved when Fe (III) is used in combination with lime (Kerslake et al., 1946). This implies that the high pH challenge associated with the use of alum as coagulant cannot be avoided when Fe (III) is used instead. However, Fe (III) stands a better chance for acceptability and wide application as Fe is less toxic than Al.

Since the combination of phytoremediation and chemical action in water decontamination is already known to increase decontamination efficiency (Braen and Weinstein, 1985; Nowack et al., 2006; Wuana and Okieimen, 2011), this study investigated the combined effect of phytoremediation (by *Amaranthus hybridus*) and Fe (III) coagulation in defluoridation of water. *Amaranthus* species was selected based on its higher fluoride bioaccumulation reported (Njega et al., 2005; Yadav et al., 2012) and higher growth rate. The motivation for this was the fact that plants roots releases dissolved CO_2 and O_2 through respiration and photosynthesis, respectively, in species which are known to effect coagulation of Fe (III) in aqueous media (Devonshire, 1890; Kerslake et al., 1946).

Experimental procedures

The fluoride stock solution (1000 mg/L) was prepared from NaF by using standard procedures using distilled water (Anonymous, 1999). Lower concentrations were prepared by standard dilution of the stock solution to obtain 5, 10, 15, 20 and 25 mgF/L concentrations using tap water. The Fe(III) stock solution of concentration 1 M Fe(III) was prepared by standard procedures using reagent grade anhydrous ferric chloride (Anonymous, 1999). Other concentrations were obtained by appropriate dilution of this stock solution. The total ionic strength adjustment buffer (TISAB 2) was prepared by standard method using 1,2 cyclohexylene-diaminetetraacetic acid (1,2-CDTA).

Experimentation

To determine the effect of time, the roots of the 10 days old amaranth seedlings were washed carefully with tap water until visually clean wash water was obtained. The roots were then blotted by a clean and dry blotting paper such that no droplets were observable in root parts. Then 9 seedlings were immersed in 50 mL of 10 mg/L fluoride solution contained in 100 mL plastic beaker in triplicate. The setting was such that only roots of the nine seedlings were immersed completely into the containers with solution. The containers with plants were exposed to the sun light and left for up to 24 h. During this time, 10 mL of fluoride water were drawn from the reactor at intervals of 12, and 24 h for fluoride analysis by pH/ISE OrionMeter fluoride meter. Standard procedures were adhered to during fluoride analysis (Anonymous, 1999).

The effect of initial fluoride concentration was determined by putting 50 mL of the solutions whose concentrations were 0, 5, 10, 15, 20 and 25 mgF/L, into different containers in triplicate. Then, the roots of about nine amaranth seedlings were immersed completely in fluoride solution in each container for 12 h. The containers were exposed to the sun light and left for observation for 12 has stated above.

In determining the effect of pH on the fluoride removal efficiency, fluoride solution with pH and fluoride concentration of 5 and 10 mgF/L, respectively was prepared and filled in the three separate 50 mL plastic containers. Nine seedlings were introduced in each separate container and left in the sun light for 12 h after which the samples were analyzed as stated above. The procedure was repeated for pH 7 and 8. The various pHs were obtained by adjusting pH using 0.1 M NaOH and HCl.

The effect of Fe(III) on the extent of fluoride coagulation and precipitation was obtained by preparing the Fe (III) solution of concentration 10 mgF/L by diluting 10 mL of 1000 mgF/L fluoride standard to 1 L using 0.1 mM $FeCl_3$ aged solution in a 1 L plastic volumetric flask. This procedure was repeated using 1 and 10 mM $FeCl_3$ aged solutions. Then, 50 mL of each solution was treated with 9 seedlings for up to 12 h undisturbed as explained above. Then, the fluoride concentration of the residue solution was analyzed as explained above. To isolate the effect of iron (III) coagulation of fluoride, parallel experiments were conducted in the absence of plants for equal amount of time as experimental

Table 1. The effect of time, initial fluoride, pH and Fe (III) on fluoride removal.

Initial F (mg/L)	FRE1 (%)	FRE2 (%)	FRE3 (%)	Average	SD	SE	r
0	0	0	0	0	0	0	0.523915
5	0	2	0	0.666667	0.942809	0.544331	
10	1	4	3	2.666667	1.247219	0.720082	
15	13.33	6.77	13.33	11.14333	3.092414	1.785406	
20	25	30	20	25	4.082483	2.357023	
25	4	0	4	2.666667	1.885618	1.088662	
Time (hours)				**Mean**			
0	0	0	0	0	0	0	-0.188982
12	2	3	3	2.666667	0.471405	0.272166	
24	0	-1	-1	-0.66667	0.471405	0.272166	
pH				**Mean**			
5	10	20	20	16.66667	5.773503	3.333333	-0.96682
7	5	0	1	2	2.645751	1.527525	
8	0	2	0	0.666667	1.154701	0.666667	
Fe(III) concentration (mM)				**Mean**			
0.1	30	20	19	23	6.082763	3.511885	0.803581
1	30	40	40	36.66667	5.773503	3.333333	
10	30	50	50	43.33333	11.54701	6.666667	

FRE = Fluoride removal efficiency, SD = standard deviation, SE = relative standard deviation, r = correlation coefficient.

reactors.

RESULTS AND DISCUSSION

When the equilibration time was increased from 0 to 12 then to 24 h, the fluoride concentration in the remnant solution showed no significant change in concentration as shown in Table 1. The slight increase in fluoride concentration observed at 24 h could be associated with the leaching out of the previously up taken fluoride from the plant. The inability of the amaranthus to uptake fluoride from the solution to the extent of reducing its concentration regardless of its reported fluoride bioaccumulation capacity (Njega et al., 2005; Khadare and Rao, 2006) could be attributed to the fact that fluoride is not among the essential elements for plant growth/survival (Tucker, 1999; Silva and Uchida, 2000). It could also imply that the 3% removal efficiency after 12 h could be due to adsorption of fluoride on root surfaces which are later released. Plant showed slight wilting during the day time but regained their vigor next morning. As fluoride initial concentration was varied from 0 (tap water), 5, 10, 15, 20 to 25 mgF/L, the fluoride removal efficiencies varied from 2 to 25% with highest removal at 20 mg/L as shown in Table 1.

When the pH was varied from 5, 7 to 8 and the reactors left undisturbed for 12 h, the removal efficiency ranged from 2 to 10, with highest removal being 10% at pH 5. This implies that fluoride removal is higher in the acidic media than in alkaline media as shown in Figure 1. This enhanced removal in acidic media could be due to the charged root surfaces (Fawell et al., 2006; Bhatnagar et al., 2011) in acidic media or increased fluoride uptake (Jagtap et al., 2012).

When the seedling roots of the Amaranths were immersed in the fluoride solution with 0.1 mM $FeCl_3$ solution, the removal efficiency after 12 h was about 20%, for 1 mM $FeCl_3$ solution, the removal efficiency after 12 h was about 37%, for 10 mM $FeCl_3$ solution, the removal efficiency after 12 h was about 40% as shown in Table 1. When fluoridated solution of 10 mM Fe (III) was tested in open air but without plants for fluoride coagulation, it was found that only 10% of fluoride was removed. Therefore, some other processes than normal Fe (III) fluoride coagulation is involved to bring about increased fluoride removal when plants were immersed in fluoridated iron (III) solutions. Results of analyses of fluoride complexed by Fe (III) in variable time indicate that over 95% of fluoride present is analyzable by 1,2 CDTA prepared TISAB 2 as indicated in Figure 3.

On the other hand, the 0.1 Mm fluoridated Fe(III)

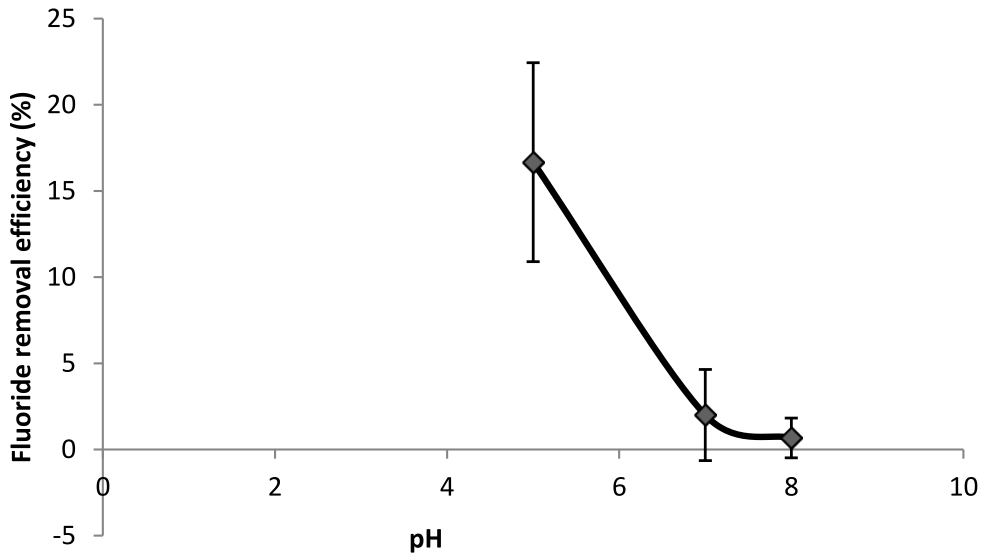

Figure 1. The effect of pH on fluoride removal by amaranth.

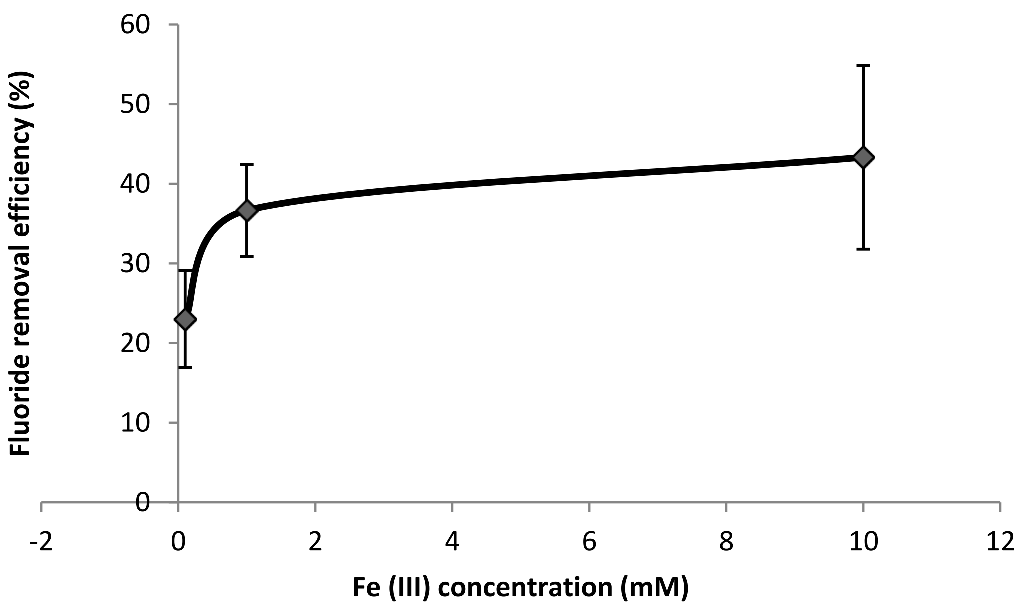

Figure 2. The effect of Fe (III) concentration on fluoride removal.

solution had pH reading of 5 at the start of experiments (Figure 2). Thus, the effect of pH could be suspected as the dominant factor for fluoride removal. However, comparison of the HCl induced and Fe (III) induced pH 5 showed that for HCl induced pH 5, only 10% fluoride removal efficiency was obtainable after 12 h while in Fe (III) induced pH 5, the removal rose to 20%. It can thus be fairly stated that the higher removal in Fe (III) is not from the effect of pH alone. The added efficiency of in Fe (III) treated fluoride may be contributed by a number of factors including plant exudates induced coagulation of Fe (III) and adsorption onto charged Fe (III) oxide film on root surfaces (Devonshire, 1890). This was supported by appearance of the solution in the plant treated reactor as compared to those that were not exposed to plants. The plant treated solution was flocculated at the end of the reaction and appeared slightly decolorized (with a decrease in absorbance from 2.88 to 1.06 at

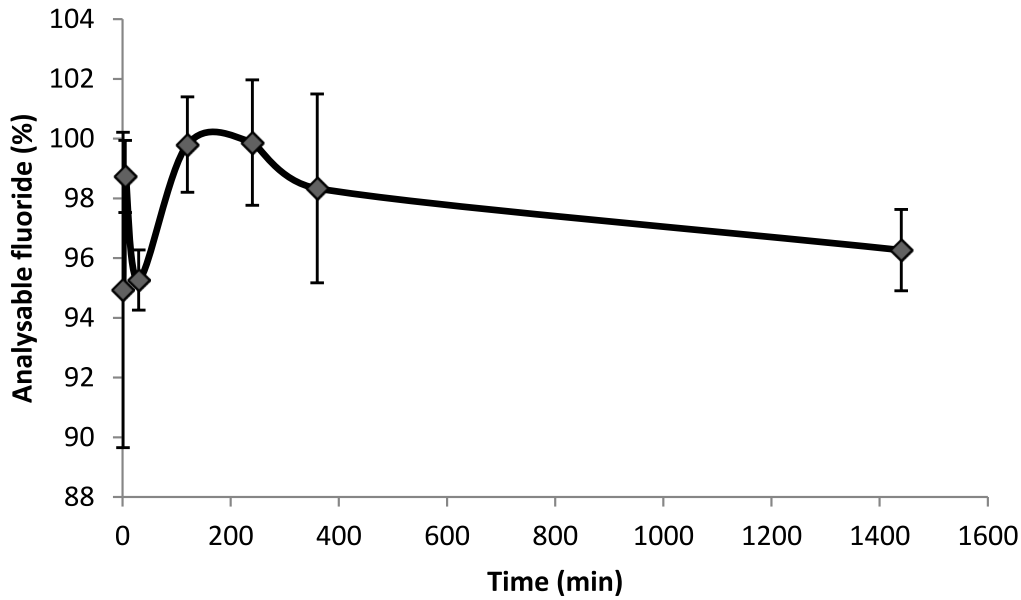

Figure 3. Effect of time on percentage analyzable fluoride in Fe (III) solution.

wavelength 425 nm) than the ones not exposed to plants.

From Table 1 and Figure 2, it is clear that there was high removal of fluoride by amaranthus when the Fe (III) was added. However, the amaranth plant wilting was higher at low pH and when Fe (III) was added. Since amaranth is not an aquatic and can thus not withstand water logging, the experiments could not be extended for times longer than 24 h.

CONCLUSION AND RECOMMENDATION

This work investigated the effect of amaranth plant roots in enhancing defluoridation efficiency of Fe (III) in batch reactors. From this work, it was found that, the peak fluoride removal efficiency at initial fluoride concentration of 10 mg/L was only 3%. Fe (III) increased this fluoride removal efficiency to about 40% with removal efficiency increasing with increasing concentration of Fe (III) in fluoride water. These findings suggest that fluoride coagulants such as Fe salts could be used in what is called chemical assisted phytoremediation of fluoride from water. Since amaranth is not an aquatic plant, further experiments are needed using a variety of aquatic plant species. It is thus recommended that further experiments be conducted using different types of aquatic plants, especially those that bioaccumulate Fe.

CONFLICT OF INTERESTS

The authors have not declared any conflict of interests.

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