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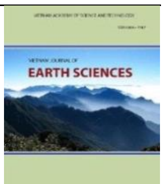

Research on the use of calcium carbide residue from the acetylene gas manufacturing factory to produce non-calcined brick

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ABSTRACT

In Vietnam, calcium carbide residue (CCR) from the acetylene gas factories are not properly treated, causing serious environmental pollution. Based on mineral composition determined by XRD, calcium carbide residue consisted mainly of portlandite (70–72%), calcite (14–16%), hydrocalumite (6-8%), and chemical composition determined by XRF method composed of CaO (53.02%), LOI (39.72%). This calcium carbide residue can be used as a source of hydrated lime, mixed with fly ash, sand, and cement to produce non-calcined bricks and test results show that brick specimens achieved compressive strength 3.0–7.5 MPa, water absorption 12.3–17.5%, density 1.28–1.80kg/cm^3. The test bricks satisfied Vietnamese standards for construction bricks.

Keywords: calcium carbide residue; non-calcined brick; fly ash; portlandite.

1. Introduction

Calcium carbide residues (CCR) are wastes from acetylene gas production by using calcium carbide. Calcium carbide is a chemical compound with the chemical formula of CaC_2. Calcium carbide is used for the preparation of acetylene gas according to the equation (1). The reaction of calcium carbide with water was discovered by Friedrich Wohler in 1962:

$$CaC_2 + 2H_2O = C_2H_2 + Ca(OH)_2 \quad (1)$$

For the preparation of acetylene gas in industries, depending on the production capacity of each factory, on an average of acetylene gas production, 64 grams of calcium carbide generates 26 grams of acetylene gas and 74 grams of calcium carbide residues in terms of $Ca(OH)_2$ (Isah and Sharmila, 2015). In the world, China is one of the countries producing a large amount of calcium carbide, accounting for 96% of the total global production, approximately 10 million tons/year and also has the amount of CCR up to 2,500 tons/year (Lui et al., 2011). In Thailand, from 2002 to 2007, in each year acetylene gas factories generate about 12,000 tons of CCR (Manasseh and Joseph, 2016). The scientists have interested in research and used CCR in many fields such as admixtures for cement (Hongfang et al., 2015; Jaturapitakkul, Roongreung, 2003; Wang et al., 2013), binder as a mix of calcium carbide residues with fly ash or rice husk ash in the manufacture of concrete (Amnadnua et al., 2013; Makaratat et al., 2010; Manasseh and

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Joseph, 2016), use of CCR to stabilized clay and laterite soil (Apichit et al., 2013; Itthikorn et al., 2017). The Middle East Gas Association has taken up this important issue in order to educate the Industrial Gases community and the public in general on the use of calcium carbide waste in the production of construction materials, improvement of agricultural land, treatment of Biosolids and Sludges, industrial sludges and petroleum wastes, animal wastes, industrial wastewater, industrial emissions such as SO_2, HCl,... (www.megases.org).

In Vietnam, during the past time, the problem of waste from acetylene gas factories and shipyard companies has not yet been treated properly and caused environmental pollution such as:

Trang Kenh Chemical and Calcium Carbide Company in Hai Phong province, the production of acetylene gas bottles from calcium carbide emitted more than 40,000m^3 of CCR and must be discharged into the company campus, leading to water pollution in the surrounding residential areas (Fig. 1) (http://baophapluat.vn).

Vietnam Industrial Gas JSC (THANH GAS) in Tien Son industrial zone, in Bac Ninh province, about 60m^3 of CCR was discharged in each month and it has not yet been treated.

Dong Anh Industrial Gas Limited Company in Ky Lien ward, Ky Anh town, Ha Tinh province in November 2016 illegally discharged 30 tons of CCR into a blank area (Fig. 2) (https://dantri.com.vn).

*Figure 1.*CCR of Trang Kenh Chemical and Calcium carbide Company in Hai Phong (http://baophapluat.vn)

Figure 2. CCR of Dong Anh industrial gas Limited Company was illegally discharged (https://dantri.com.vn)

Hyundai Vinashin Khanh Hoa Shipyard Co., Ltd dumped calcium carbide residues into the landfill site of Long My Waste Management and Processing Enterprise, in Phuoc My commune, Quy Nhon City, Binh Dinh province (Fig. 3a, b) (https://tuoitre.vn).

All of these cases can pollute the environment and cause annoyance to local people.

In general, sources of CCR from acetylene gas factories in Vietnam are being dumped into the storage tanks at these companies or illegally buried without the right treatment solution. This causes environmental pollution on the one hand and waste of useful raw materials on the other hand.

Before the above reality and the accumulation of experience in the production of non-calcined bricks from artificial and natural pozzolans (Nguyen Anh Duong, 2011; Nguyen Anh Duong et al., 2014; Kieu Quy Nam and Nguyen Anh Duong, 2010), based on the results of the full study of the mineral and chemical composition of calcium carbide residues, the authors offer a solution to making this waste source into a useful material. That is a mixture of calcium carbide residues with activated pozzolan materials such as fly ash to produce non-calcined bricks.

Figure 3. a-A staff of Centre for Natural Resource and Environmental Monitoring took CCR samples at Long My landfill site, in Binh Dinh; b-CCR of Hyundai Vinashin Khanh Hoa Shipyard Co., Ltd dumped into the landfill site of Long My Waste Management and Processing Enterprise

2. Materials and Methods

2.1. Materials

Materials used in the study are calcium carbide residues of Vietnam Industrial Gas JSC (THANH GAS) in Tien Son Industrial Park, Bac Ninh province, fly ash of Pha Lai Thermal Power JSC, the cement of VICEM BIMSON Cement JSC and natural sands.

2.2. Methods

To study and assess the mineral and chemical composition of CCR, fly ash, and test bricks, research methods were used as follows:

- Semi-quantitative analysis of minerals composition: The mineralogical analysis was carried out by X-ray diffraction (XRD) on the Empyrean (PANalytical, Netherlands) system in the Institute of Geology Sciences, Vietnam Academy of Science and Technology (IGS-VAST) with a Cukα source at 45kV and 40mA. Mineral phases were identified in comparison with the PDF-2 database, after separating and semi-quantifying diffraction peaks by the HighScore Plus 4.5 software.
- The chemical composition analysis: The chemical composition of CCR and fly ash was determined by X-ray fluorescence (XRF) on S4 - Pioneer system at IGS-VAST.
- Test of physicomechanical properties: water absorption and bulk density were determined according to TCVN 6355-4: 2009 standards and TCVN 6355-5: 2009 standards, respectively, at IGS-VAST. The compressive strength of the samples was measured according to TCVN 6477:2011 standard at the Institute for Building Materials, Ministry of Construction.
- Process for preparing non-calcined bricks: Non-calcined bricks were prepared by mixing calcium carbide residues, fly ash, natural sand, and cement at different ratios (table 3). These mixtures were added water, then filled in steel mounds and compressed with the static pressure 30kG/cm^2.

3. Results

3.1. Mineralogical and chemical characteristics of calcium carbide residues

The main mineral composition of calcium carbide residues used in this study includes: portlandite (70–72%), calcite (12–14%), hydrocalumite (5–7%), few minerals such as quartz and gypsum (Fig. 4, Table 1). The main chemical composition of calcium carbide residues was CaO (53.02%), SiO_2 (3.65%) and Al_2O_3 (2.16%), the detrimental chemical component such as SO_3 are negligible (Table 2).

Table 1. Mineralogical compositions of CCR and fly ash(by XRD analysis)

Mineral component (~%, by mass)	Sample	
	CCR	TB
Portlandite	70-72	-
Calcite	14-16	-
Quartz	3-5	14-16
Mullite	-	15-20
Hydroclumite	6-8	-
Gypsum	5-7	-
Carbon	-	5-7
Hematite	-	minor
Amorphous	-	67-73

3.2. Mineralogical and chemical characteristics of fly ash

The major mineral composition of fly ash (TB) of Pha Lai Thermal Power JSC included: Mullite (15–20%), quartz (14–16%), carbon (5–7%), and little hematite (Table 1). Besides the composition of the crystalline phases, on the XRD diagram (Fig. 4) at the angle of 15–40° 2-theta a hump was created, that shows that fly ash contained a large amount of amorphous component. Results of quantitative XRD analysis of amorphous content in fly ash about 67–73%. The chemical composition of fly ash was mainly SiO_2 (51.73%) and Al_2O_3 (23.22%) (Table 2).

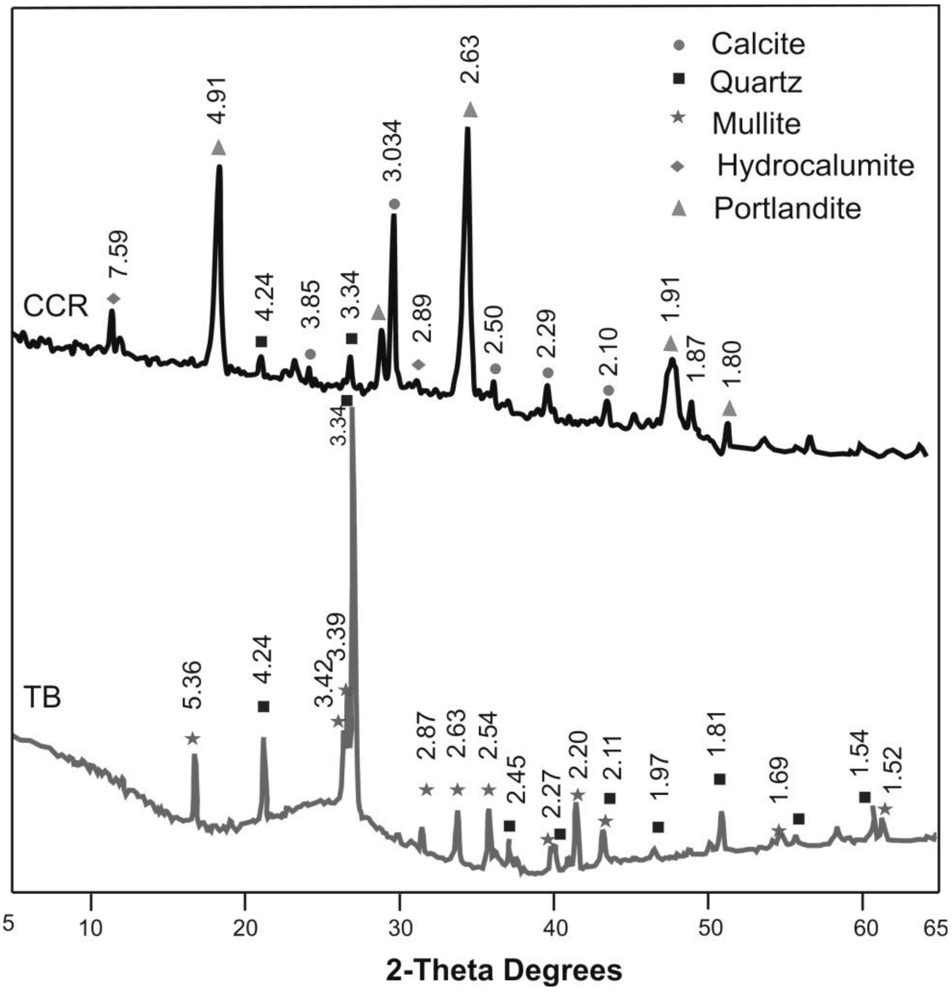

Figure 4. The XRD diagram of calcium carbide residues (CCR) and fly ash (TB)

Table 2. Chemical compositions of calcium carbide residues and fly ash

Component (%)	Sample	
	CCR	TB
SiO_2	3.65	51.73
TiO_2	0.03	0.94
Al_2O_3	2.16	23.22
T-Fe_2O_3	0.12	4.23
MnO	<0.01	0.05
MgO	0.17	1.52
CaO	53.02	0.89
Na_2O	<0.01	0.09
K_2O	-	4.49
P_2O_5	<0.01	0.14
SO_3	0.21	0.44
Cl	0.06	-
LOI	39.72	11.26

3.3. Test results of non-calcined bricks

As presented in the previous articles (Nguyen Anh Duong, 2011; Nguyen Anh Duong et al., 2014, Kieu Quy Nam and Nguyen Anh Duong, 2010), in principle, the combination of pozzolan with hydrate lime will form calcium-silicate-hydrate (CSH), calcium aluminate silicate hydrate (CASH) phases like the binders formed in hydration cement. The above analysis results showed that the main component of CCR was hydrate lime so it was a kind of binder. Fly ash is known to be a highly activated pozzolan. Therefore, we used the CCR as a binder or combining two sources of industrial wastes (CCR and fly ash) to produce non-calcined bricks following themethod "pozzolan + lime".

The specimens of non-calcined bricks were tested and fabricated by mixing of calcium carbide residues: fly ash: natural sand: cement at different proportions shown in Table 3. These mixtures were mixed together, after that moistened with 12–14% water by weight, and then filled into steel mounds and compressed with the static pressure 30kG/cm^2 to form a brick specimen with a cylindrical shape (5cm × 5cm in size) (Fig. 5). Specimens were cured by water-saturated covers at room condition. After 28 days, these specimens were determined physicomechanical properties such as compressive strength, water absorption, and bulk density.

Table 3. Mix proportions of calcium carbide residues with other materials

Test Designation	Mix proportion (%)			
	CRR	Sand	Fly ash	Cement PCB30
M1:1	50	0	50	0
M5:3	40	30	30	0
M4:2	50	30	20	0
M2:1	65	35	0	0
M2:2	50	50	0	0
M1	80	10	5	5
M2	75	10	10	5
M3	70	10	15	5
M4	75	15	5	5
M5	70	20	5	5
M6	70	20	10	0
M7	80	10	10	0
M8	80	10	0	10
M9	75	20	0	5
M10	60	30	0	10
M11	60	30	10	0

Figure 5. Specimens of non-calcined bricks were produced from CCR

Test results of products showed that compressive strength was from 1.3 to 7.5MPa, water absorption was from 12.3 to 20.8%, the bulk density of products was quite light ranging from 1.28 to1.80 kg/cm^3 (Table 4).

Table 4. Physico-mechanical properties of non-calcined bricks produced from CCR

Test Designation	Bulk density (kg/cm^3)	Water absorption (%)	Compressive strength (MPa)
M1	1.25	21.0	2.8
M2	1.80	12.3	4.3
M3	1.81	12.5	4.5
M4	1.30	20.5	3.2
M5	1.46	19.5	3.6
M6	1.43	13.6	3.8
M7	1.42	17.5	3.0
M8	1.44	17.6	4.7
M10	1.28	20.3	5.3
M1:1	1.53	15.0	6.3
M11	1.70	15.1	3.0
M5:3	1.70	13.2	7.5
M4:2	1.72	14.5	6.2
M2:1	1.54	21.3	1.3
M2:2	1.37	20.8	1.4
TCVN 6477-2011(Standard for Concrete brick)	<1.65	6-18	3.0-20

4. Discussions

The results of the above analysis showed that the main component of calcium carbide residue was portlandite, which was a hydrated lime. Therefore, this material is a low-grade binder, on the other hand, it can be used as an alkali activator which can combine with pozzolan materials to form calcium-silicate-hydrate (CSH), calcium aluminate silicate hydrate (CASH) binders (Bui Van Chen and Dao Tien Dat, 1985; Nguyen Anh Duong, 2011; Nguyen Anh Duong et al., 2014).

In addition, calcium carbide residues contained a number of minerals including calcite and hydrocalumite.

Hydrocalumite mineral exists in CCR because the production technology of calcium carbide used raw materials such as limestone and coke. In addition to the main components including carbon in coke and calcium in limestone, the raw materials also contained an amount of Al_2O_3 and SiO_2 content (Table 3). When limestone was calcined with coke at 2200°C, CaC_2 was created. In this process, a part of CaO combined with Al_2O_3 to form Monocalcium aluminate ($CaAl_2O_4$) at 1390°C, which is similar to the binders in aluminate cement (Matschei at el., 2007). Monocalcium aluminate reacts to water in order to form a hydrated compound with the formula of $3CaO.Al_2O_3.6H_2O$. This compound combines with $Al(OH)_3$ and $Ca(OH)_2$, $CaCl_2.2H_2O$, $AlCl_3 \cdot 6H_2O$ and water in ambient temperature conditions to form "Friedel" salt ($3CaO.Al_2O_3.CaCl_2.10H_2O$) or hydrocalumite ($Ca_4Al_2(OH)_{12}Cl_2.6H_2O$) (Jame et al., 2004; Jiao and Qinghai, 2014).

Calcite phase in calcium carbide residue was formed by the reaction of portlandite with CO_2 gas in the natural environment to form calcium carbonate.

Fly ash of Pha Lai Thermal Power JSC had a main component of SiO_2 and Al_2O_3. A small part of this compound existed in the crystalline phase as quartz and mullite, the majority was amorphous SiO_2 and Al_2O_3. According to Ludwig and Schwiete (1963) (in Mustafa Tokyay, 2016), glass components were capable of combining with a significant amount of lime reaching 364mgCaO/g. In fly ash used in this study, amorphous content reached 67–73%, thus 100 grams of fly ash can combining-lime to 24.4–26.6g CaO. Therefore, to completely react with the amount of $Ca(OH)_2$ in the calcium carbide residue, it needs a certain amount of amorphous compound in fly ash, and the maximum mixing ratio between CCR and fly ash was 1:2. This amorphous compound creates pozzolanic activity of fly ash. When it combined with hydrated lime, it will create the same binder phase as cementitious to make strength and water resistance for construction materials producing from this material.

The results of the physico-mechanic properties of non-calcined bricks producing from calcium carbide residue and sand showed that the specimens had a light bulk density (1.37–1.54kg/cm^3). This light bulk density is a beneficial factor for construction because it helps decrease the weight of the foundation and reduce construction costs. However, with the light bulk density, the porosity and water absorption of the bricks often increase. The evidence for this is the experiment on brick production in this research, the study specimens had quite high water absorption (from 20.8–21.3%).

In brick specimens, calcium carbide residue was used as binders and sand played the role of aggregates. The hardening process has created the compressive strength of non-calcining brick specimens including Firstly, the condensation of colloids and crystallization of $Ca(OH)_2$ make the crystals come near and bind together; The second is the combination with CO_2 in the air, the carbonation process takes place in equation (2) to form crystalline calcite phases. The alternate formation of crystalline $CaCO_3$phases and $Ca(OH)_2$ crystals makes the bricks become hardened. Results of XRD analysis clearly showed portlandite phase in calcium carbide residue with peaks at angles of 18.09; 28.69; 34.09; 47.12; 50.8; 54.35 2-theta and d values of 4.91; 3.11; 2.63; 1.93; 1.79; 1.68 Å, respectively were transformed into calcite phase in non-calcined bricks with peaks at 29.44; 36.02; 39.46; 43.22; 47.58; 48.58 2-theta and d values of 3.03; 2.49; 2.28; 2.09, 1.91; 1.87 Å, respectively (Fig. 6).

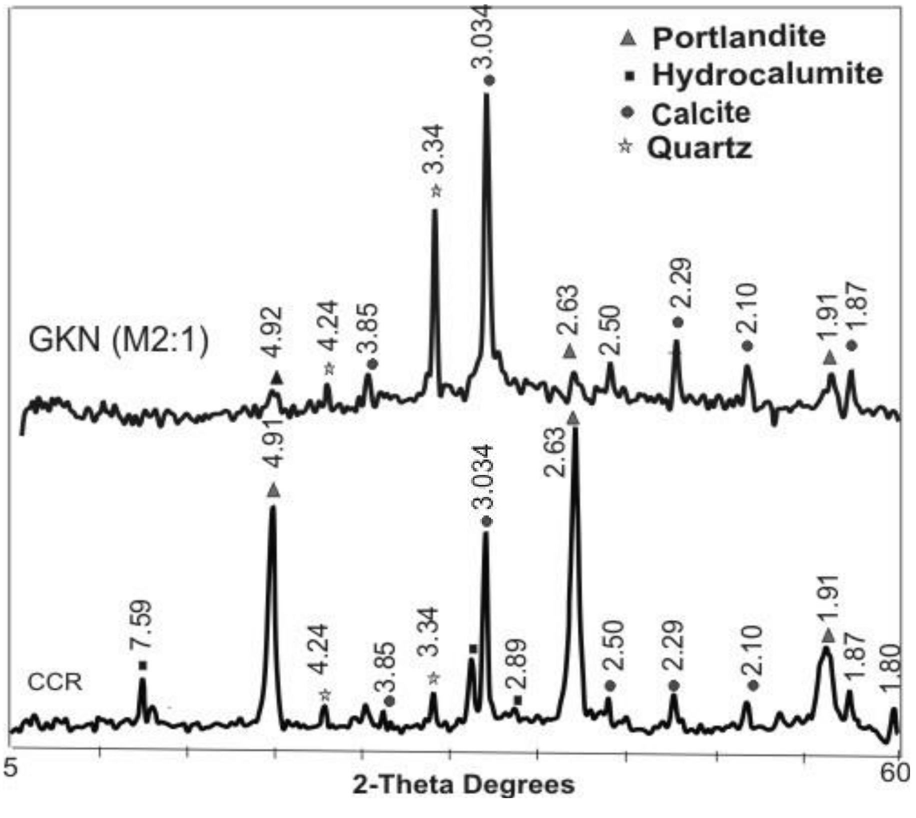

Figure 6. TheXRD diagram of non-calcined brick producing from the mixture of calcium carbide residue and sand (GKN), and calcium carbide residue (CCR)

$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O \qquad (2)$$

The specimens produced according to this method, after 28 days, usually have a low compressive strength such as the specimen M2:1 with a mixing ratio of 50% calcium carbide residue and 50% sand, compressive strength of 1.3Mpa, and the specimen M2:2 with a mixing ratio of 65% calcium carbide residue and 35% sand, compressive strength of 1.4Mpa, water absorption of more than 20%. The compressive strength of the products was not high because the carbonation process occurs in natural conditions relatively slowly and the calcite phase is a binder with low compressive strength (Bui Van Chen, Dao Tien Dat, 1985). Therefore, non-calcined bricks produced from the mixture of calcium carbide residue and sand do not satisfy the technical requirement for construction bricks.

In order to increase the compressive strength of products, it is necessary to add a high-grade binder such as cement or pozzolanic admixtures, for example, fly ash to form a binder similar to cementitious value.

For test specimens using fly ash in the mixture, their compressive strength was higher than the specimens which were not added fly ash. Because in addition to the binding phase which was calcite ($CaCO_3$) and formed by equation (1), in the samples, amorphous, semi-crystalline to crystalline CSH and CASH binding phases were formed by the combination of hydrated lime $Ca(OH)_2$ and amorphous compounds in fly ash such as Al_2O_3, SiO_2 according to the equation (3-5) (Bui Van Chen, Dao Tien Dat, 1985; Nguyen Anh Duong et al., 2014).

$$nSiO_2+Ca(OH)_2 \rightarrow nCaO.SiO_2.xH_2O(CSH) \quad (3)$$

$$nAl_2O_3+Ca(OH)_2 \rightarrow nCaO.Al_2O_3.xH_2O(CAH) \quad (4)$$

$$nCaO.Al_2O_3.xH_2O+SiO_2 \rightarrow nCaO.Al_2O_3.SiO_2.xH_2O \ (CASH) \quad (5)$$

According to the researches by Lui et al. (2004), Jeffrey et al. (2002), William (2013), Ian (2014), d values of the diffraction peaks of CSH phases were 9–14; 5.3; 4.36; 2.1; 2.4; 2.78–2.97; 3.2; 3.6; 3.03–3.07Å and 1.80–1.83Å. Results of XRD analysis showed that d values of the diffraction peaks of the study specimen at 3.03; 2.49; 2.84; 2.88Å coincided with the diffraction peaks of calcite phase and d values of the diffraction peaks at 5.39, 2.12, 1.82Å coincided with the diffraction peak of the mullite and no diffraction peak at 9–14Å were observed (Fig. 7). This proves that at laboratory temperature, CSH and Al-CSH phases exist in amorphous to semi-crystalline form and cannot be transformed into a complete crystallize phase as tobermorite.

The results of physicomechanical properties showed that the brick specimens added fly ash in the mixture reached compressive strength from 3.0–3.8Mpa (specimens: M6, M7, M11) to 6.3–7.5MPa (specimens: M1: 1, M5: 3, M4: 2). From the obtained analytical results, it can be seen that when the amount of fly ash increases, the compressive strength of the bricks also increases, and the water absorption decreases remarkably. Because during the brick formation, fly ash will play the role of a pozzolanic material and the calcium carbide residue is an alkaline agent, they combine together to form high-grade binders like CSH, CASH. For test specimens added sand in the mixture, the compressive strength of the test brick specimens increases and water absorption decreases. This was also proved by Phung Van Lu et al. (1999), according to this author, sand was considered as a fine aggregate when it was mixed at a reasonable ratio, it will reduce porosity and contribute to increasing the strength of the bricks.

Instead of using a large mass of fly ash from 20–50% down to 0–15% and a small amount of cement (5–10%) was used to promote the hardening process more quickly and still ensure the strength. The results of brick specimens used 5–10% of cement and 10–15% of fly ash reached the compressive strength of 4.3–5.3Mpa (specimens: M3, M4, M8, M10).

However, with the purpose of using as much calcium carbide residue as possible in the mix proportion, the appropriate proportions (calcium carbide residue : sand: fly ash: cement) were M6-70:20:0:0 and

M2-75:10:10:5. In the region where both raw material sources including calcium carbide residue and fly ash are available, it should be used a mixture with the high ratio of fly ash content (specimens M5:3 and M4:2) to produce high-quality bricks.

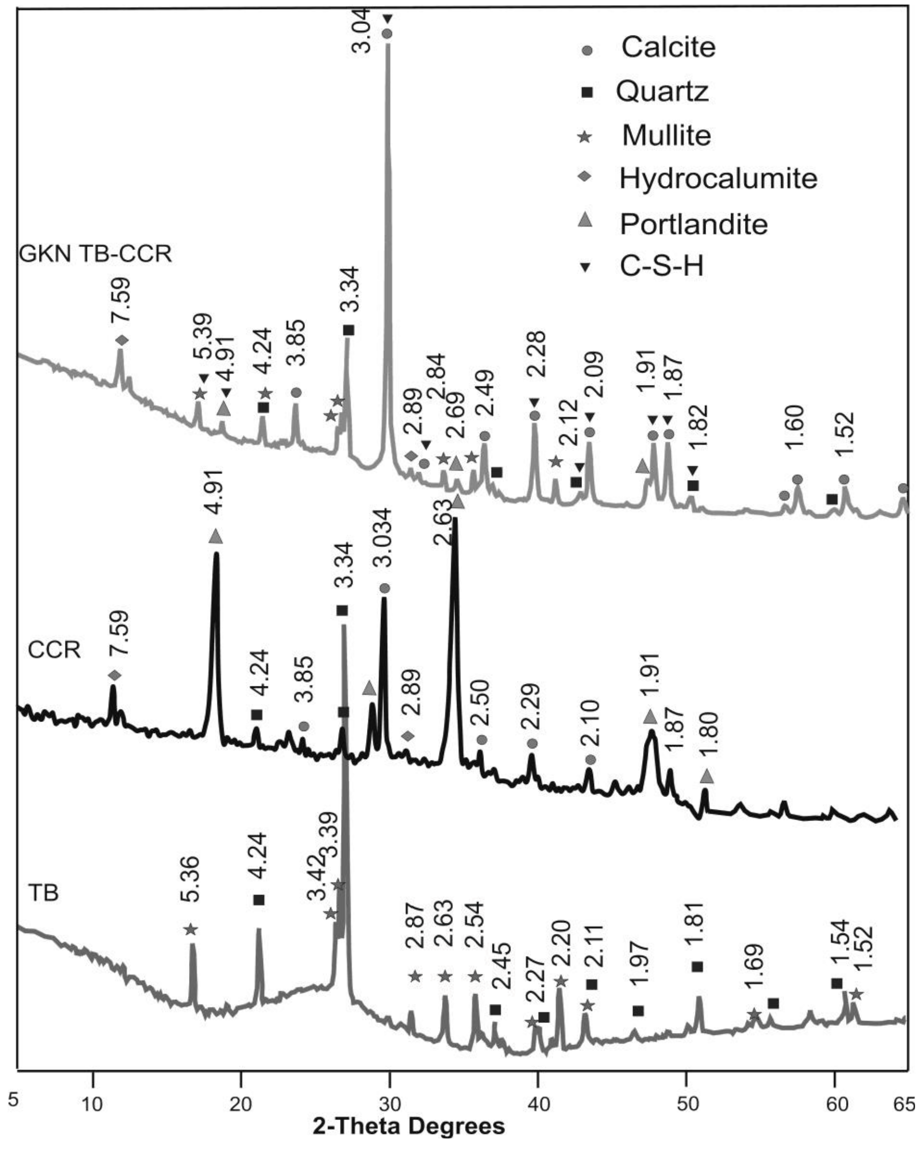

Figure 7. The XRD diagram of non-calcined brick produced from the mixture of calcium carbide residue and fly ash (GKN -TB- CCR); calcium carbide residue (CCR); Fly ash (TB)

5. Conclusions

(i) Calcium carbide residue had portlandite content (70–72%), calcite (10–12%), hydrocalumite (6–8%), quartz and gypsum. Calcium carbide residue from the acetylene gas factories is a good quality hydrated lime source to use in construction material production.

(ii) Non-calcined bricks are produced from a mixture of calcium carbide residue and sand had low compressive strength (1.3–1.4Mpa) and high-water absorption (20.8–21.3%).

Therefore, they do not satisfy the technical requirements for construction bricks.

(iii) With the mix proportion used 40–80% of calcium carbide residue, 10–50% of fly ash, 0–30% of sand and 0–10% of cement, the non-calcined bricks reached the compressive strength of 3.0–7.5Mpa, water absorption of 12.3–17.5%, bulk density of 1.28–1.80kg/cm^3. These non-calcined bricks satisfied the technical requirements for construction bricks.

(iv) The most reasonable mix proportions for tested bricks produced from calcium carbide residue of Vietnam Industrial Gas JSC (THANH GAS) (calcium carbide residue: sand: fly ash: cement) were M6-70:20:10 and M2-75:10:10:5. These mix proportions ensure the quality of the non-calcined bricks which completely satisfy the technical requirements as well as use the largest mass of calcium carbide residue as raw materials to produce non-calcined bricks.

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