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Ceramics International 35 (2009) 813–819

CERAMICS INTERNATIONAL

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Multi-impregnating pitch-bonded Egyptian dolomite refractory brick for application in ladle furnaces

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Received 5 June 2007; received in revised form 21 January 2008; accepted 22 February 2008
Available online 28 June 2008

Abstract

A method of preparation of multi-impregnated pitch-bonded Egyptian dolomite refractory brick for ladle furnace is described. Brick samples were prepared from blend of calcined dolomite mineral and coal tar pitch. The blend was hot mixed and pressed under a compression force up to 151 MPa. Green bricks were baked for 2 h at temperatures up to 1000 °C. Voids in the baked bodies were filled with carbon by multiple impregnations using low-softening point coal tar pitch. Each impregnation step (30 min) was followed by calcination at 1000 °C. Brick samples containing 8–12 wt.% coal tar pitch binder and pressed under 108–151 MPa acquired quantify crushing strength. However, multi-impregnating favored the mechanical strength of the baked brick samples and improved their hydration resistance (>45 days). Dolomite brick samples containing 10 wt.% coal tar pitch and pressed at 108 MPa gave high hydration resistance (more than 60 days in normal condition) compared to the hydration resistance of the commercial bricks (30 days). The prepared brick samples have acceptable density, chemical stability, outstanding resistance and good mechanical properties would meet the requirements of Ladle furnace (LF) for steel making industry. In addition, estimation of production cost of the brick indicates it is competitive with the market price based on durability and service life time aspects.

Keywords: Dolomite brick; Carbon impregnation; Refractory brick; Pitch-bonded brick

1. Introduction

Application of natural dolomite mineral is mainly confined to the linings of converters, ladle furnaces, various vessel types and electric arc furnaces in the steel industry [1,2]. The consumption rate of dolomite brick depends upon the working conditions such as temperature, slag composition, tap to tap time and nature of the used flux. It has also been reported that physical properties of dolomite brick could be improved by using pitch, tar, flake and vein graphite minerals [1,3]. New types of carbon-bonded dolomite brick have been prepared to meet specific service conditions [4,5] and to resist the harmful effects of slag. Such improvements have been achieved by incorporating various proportions of magnesia or carbon (up to 10 wt.%) but the cost increased.

Different oxidation treatments have been studied to increase the softening point of a commercial coal tar pitch [6]. It was shown that oxidation within the temperature range of 160–250 °C increased both the toluene insoluble and quinolin insoluble fractions and the softening point of the treated pitch. The yield of the modified pitches was considerable with H_2SO_4, H_2O_2 and HNO_3 compared to air blowing. In another study [7], coal tar pitch was modified by increasing the time up to 6 h at 430 °C. The pitch was treated by air blowing at 275 °C for 10–30 h. Blanco et al. [7] concluded that thermally treated pitches still contained a considerable amount of light compounds together with the large planar molecules generated. Light components polymerized to a considerable extent during air blowing to form cross-linked molecules. Extensive removal of aliphatic hydrogen also took place. However, the predominant reaction giving rise to the hardening of the pitch was stripping of volatile matter during thermal oxygen or hydrogen gas sparging [8]. Modification results were ascribed to either an oxidative molecular growth mechanism or incorporation of

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0272-8842/$34.00
doi:10.1016/j.ceramint.2008.02.017

oxygen functionality into the pitch and a consequential reduction of volatility. In another investigation [9], polymerization dominated volatilization of low molecular weight compounds in the pitch.

Thermal treatment of coal tar based mesophase pitch increased the compressive strength of carbon form by 64%, 96% and 100% by mixing the pitch with 2%, 5% and 10% montmorillonite clay [10]. Due to the high thermal insulation and lamellar clay structure, the thermal conductivity decreased from 2 W/m K to 0.25 W/m K. On the other hand [11], matrices parts made of the filler/pitch-based cokes showed fine mosaic microstructures. The flexural structure was significantly increased (12–15%) by the addition of the filler.

The percentage change in graphite properties due to impregnation has been reported to increase as the weight introduced into the pores goes up [12]. However, the impregnant is distributed by capillary forces in the pores [13]. Other studies [14–16] found that multiple impregnations were more efficient due to charring of the impregnant with subsequent precipitation of hard carbon in the pores. Multi-impregnation for carbon bodies increases the mechanical properties and lifetime by a factor of 4.30 [17]. For single impregnation, epoxy resin yielded the largest improvement compared to coal tar pitch. Properties such as viscosity, adhesion force and coking value determined the effectiveness of the impregnant.

The objectives of this work are to prepare brick made of pitch-bonded dolomite having good resistance to hydration, chemical stability, mechanical strength and low cost of preparation.

2. Experimental

2.1. Materials

Sintered dolomite a sample weighing about 50 kg and packed in polyethylene bag was supplied from the Egyptian Iron and Steel Company (Helwan, Egypt). The dolomite size was from 0 mm to 10 mm. The average chemical composition of this material, characterized by XRF technique, is given in Table 1. The sintered dolomite used in the study was the local raw dolomite of Adabyia Formation South Suez Town that thermally treated at Helwan plant for the production of tar-bonded dolomite blocks.

Table 1
Physical properties and chemical analysis of doloma produced at Helwan Steel Plant

Property	Sintered dolomite
Physical properties	
Apparent porosity (%)	6.87
Bulk density (g/cm^3)	2.96
Chemical analysis (wt.%)	
SiO_2	1.25
R_2O_3	5.33 [Fe_2O_3: 5.10, Al_2O_3: 0.23]
CaO	9.32
MgO	33.44
Loss on ignition	0.58

The mineralogy of this material was extensively studied using X-ray diffraction technique [18,19]. It was almost composed mainly from free lime (CaO), magnesia (MgO) and small amounts of dicalcium ferrite ($2CaO{\cdot}Fe_2O_3$) and tricalcium silicate ($3CaO{\cdot}SiO_2$).

Coal tar was made up by mixing high carbon-residue pitch with low naphthalene anthracene oil. A similar procedure of preparation of tar for the production of tar-bonded dolomite blocks at Helwan plant was applied. It was supplied by El-Nasr Coke Company (Helwan, Egypt). The data are tabulated in Table 2.

2.2. Experimental procedure

2.2.1. Brick-making

Sintered dolomite was classified into three fractions termed coarse (10–3 mm), medium (3–1 mm) and fine fraction (<0.1 mm). Three batches were prepared from blends of sintered dolomite mineral and variable tar weight ratio as given in Table 3. Brick materials were preheated at 100 °C before use. Coarse (10–3 mm) and medium fractions (3–1 mm) were first blended and part of the tar dose was then added. The ingredients were hot mixed for 5 min. The fine dolomite fraction was then added while hot mixing. The remaining dose of the tar was added and the mass so composed was hot mixed for 10 min. The mixture was then transferred to a preheated steel mould. Cylindrical-shaped briquettes of 5 cm diameter and 5 cm height were formed under a pressure of 65 MPa, 108 MPa and 151 MPa. The briquettes so formed were placed in rectangular SiC crucibles, covered with carbon/sand mixture and charged in a furnace for calcinations at 1000 °C. The heating regime was 0.5 °C/min up to 600 °C followed by a higher rate 2 °C/min up to the final temperature (1000 °C). The soaking time at the final temperature was about 2 h. After cooling to the room temperature, the brick samples were discharged and freed from dust and weighed. These were multi-impregnated using a low-softening point tar.

Each single impregnation was carried out by placing in a vacuum for 15 min followed by dipping in molten pitch under 500 kPa followed by centrifugation at 3000 rpm while. The

Table 2
Coal tar pitch properties

Property	Tar
Specific gravity, 25 °C	1.26
Viscosity (standard viscometer, cup 10 mm, 60 °C, 50 ml), s	22
Naphthalene, %	124
Pitch (>360 °C), %	76.20
Water (50 ml xylene/50 ml benzene, 2 h), %	Nil
Distillation range:	
Up to 210 °C, %	0.15
210–300 °C, %	4.18
300–360 °C, %	18.20
Softening point of pitch (Ball and Ring test), °C	82
Conradson carbon-residue test	
Residue on tar as received, %	39
Residue on pitch as made, %	50

Table 3
Body compositions

Mix no.	Composition (wt.%)			
	Sintered dolomite			Tar
	Coarse (10–3 mm)	Medium (3–1 mm)	Fine (−0.1 mm)	
1	36.8	9.2	46	8
2	36	9	45	10
3	35.2	8.8	44	12

impregnated sample was then calcined at 1000 °C in a chamber furnace to pyrolze the impregnant.

Densification parameters in terms of bulk density and apparent porosity of samples were determined according to JIS R2205. The total porosity of the brick samples was determined by a mercury porosimeter fitted with a micrometrics pore sizer 9310 U.S.

Mechanical strength in term of cold crushing strength (CCS) was carried out for three bars/measurement using Control Hydraulic Press model (Controls (CE), Datamatic, Italy).

The chemical property in term of softening point (°C) was determined according to the Kramer and Sarnow (K&S) scale using ball and ring technique. Volatile content was determined gravimetrically. The hydration resistance of the brick samples was characterized by the exposure the test specimen to atmosphere till completely collapsed or failure.

3. Results and discussion

Tables 1 and 2 show the properties of the minerals used in this study for preparation of the brick samples; the dolomite mineral and the coal tar pitches.

Fig. 1 shows the extent of volatile matter expelled upon heating the coal tar pitch for periods up to 420 min at temperatures up to 350 °C without gas blowing. It can be seen that the extent of volatiles increases with increase in temperature and time attaining maximum values ≥360 min. The maximum extent of volatiles amounts to 32% by weight after heating the pitch at 350 °C for 420 min. Fig. 2 show the effect of modification time on the softening point of pitch samples after heating at temperatures up to 300 °C. Experiments involved subsequent blowing of air or oxygen gas at a volume flow rate 250 mL/min/kg pitch. It can be seen that the softening point of the pitch samples increases linearly with modification time. Oxygen gas was more aggressive compared to air in increasing the softening point of the treated pitches. Fig. 3 shows the softening point of the modified pitch samples using nitrogen dioxide gas at 300 °C for periods up to 180 min. The curves obtained have the same shape given in Fig. 3 except that the softening point is higher with nitrogen dioxide. Fig. 4 illustrates that the aggressiveness of the examined gases was in the order NO_2, O_2 and air. It may be worthy to note that the NO_2-modified pitches were glassier, brittle and fragile compared to oxygen or air-modified pitches. Results proved that the modification removes low boiling point resins in the pitch with subsequent polymerization of some aromatic resins. The modified pitch acquired higher molten viscosity. Modification process was found to improve the properties of the pitches as well as the composites so prepared. This finding is in line with the conclusions of previous work [20,21].

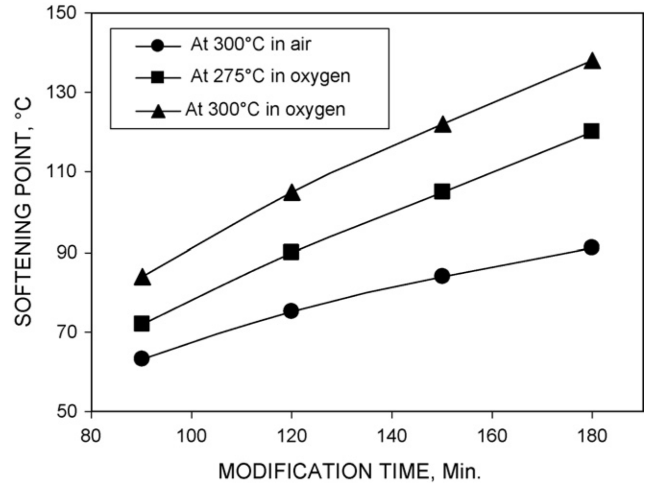

Fig. 2. Effect of modification time on softening point of treated coal tar pitch using air or oxygen gas at temperature up to 300 °C (stirring at 500 rpm, volume flow rate 500 mL/min/kg pitch).

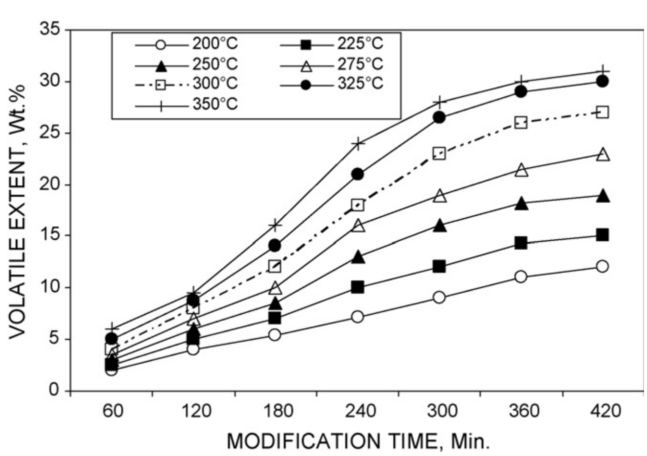

Fig. 1. Effect of modification time on extent of volatiles upon heating coal tar at temperatures up to 350 °C for 3 h without gas blowing.

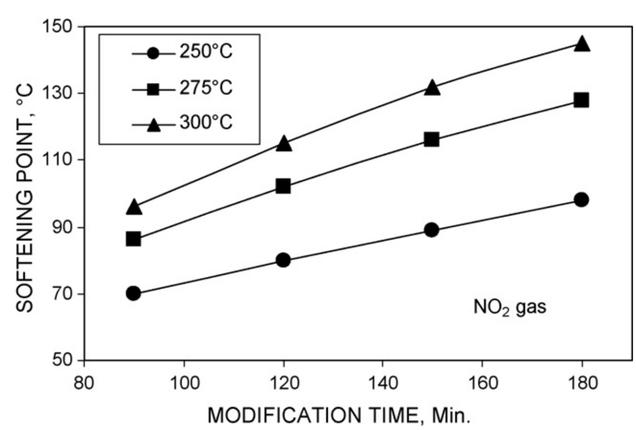

Fig. 3. Effect of modification time on softening point of treated coal tar pitch using nitrogen dioxide gas at temperatures up to 300 °C (stirring at 500 rpm, volume flow rate 500 mL/min/kg pitch).

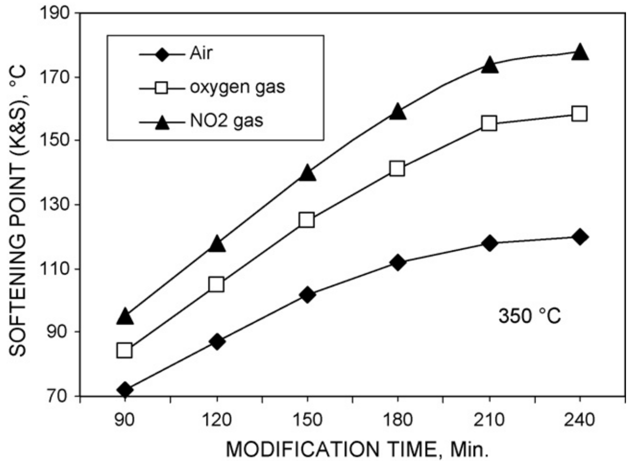

Fig. 4. Effect of modification time on softening point of treated coal tar pitch using air, oxygen and nitrogen dioxide gas at temperatures up to 300 °C (stirring at 500 rpm, volume flow rate 500 mL/min/kg pitch).

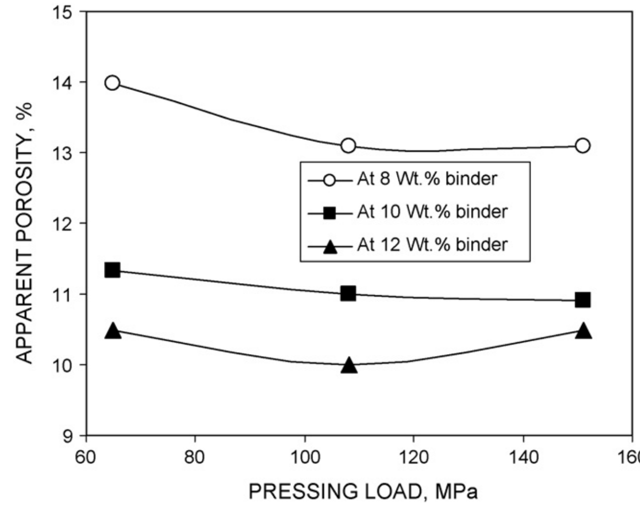

Fig. 6. Effect of pressing load on total porosity of calcined un-impregnated brick samples with different contents of coal tar pitch binder.

Fig. 5 shows the apparent density value of green dolomite bricks prepared from modified pitches as a function the pressing load. The apparent density value increases with increase in pressing load passing through a maximum at 108 MPa. This effect is more pronounced in higher binder content samples. Fig. 6 shows the total porosity of calcined and un-impregnated bricks as affected by the pressing load. The extent of porosity decreases with increase in pressing load approaching a constant value of 10.5–13% at >108 MPa. However, brick samples made of 12% pitch content and pressed at 108 MPa kg/cm^2 show the minimum porosity value (10.5%). Brick samples containing less binder (8%) acquired the highest porosity (13%). Fig. 7 shows the apparent density of fired dolomite brick samples before impregnation. The density value curves are a mirror image of that of the porosity values. Brick samples made of 12% pitch binder acquired the highest density value compared to those made of lower binder content. It is worth noting that the density value of these brick samples decreases slightly with increasing pressing load >108 MPa.

The method applied for brick preparation involved hot pressing of the ingredients. Optimum requirement of the pitch binder (10% by weight) would satisfy the intra-particle voids of the dolomite grains. Pressure exerted during brick shaping helps the molten binder to creep on the surface of the dolomite grains to fill the voids. Higher pitch content is not in favor of the brick strength as extra quantity would be squeezed upon pressing. Squeezing of the molten binder outside the brick body would cause less binding material still remaining in the brick body. It becomes therefore legitimate that the formed carbon framework inside the brick body decreases leading to a corresponding decreases in the mechanical strength.

Fig. 8 shows the residual carbon values of dolomite brick samples having different contents of coal tar pitch binder as a function of the pressing load. The residual carbon increases with increasing the coal tar pitch dose and pressing load. It is also noted that increasing the pressing load from 108 MPa to 151 MPa does not bring about a significant change in the residual carbon of the investigated batches. On the other hand, increasing the coal tar content and pressing load as given in Table 4, improved the hydration resistance in terms of time required to cause failure of the dolomite brick samples. Brick samples

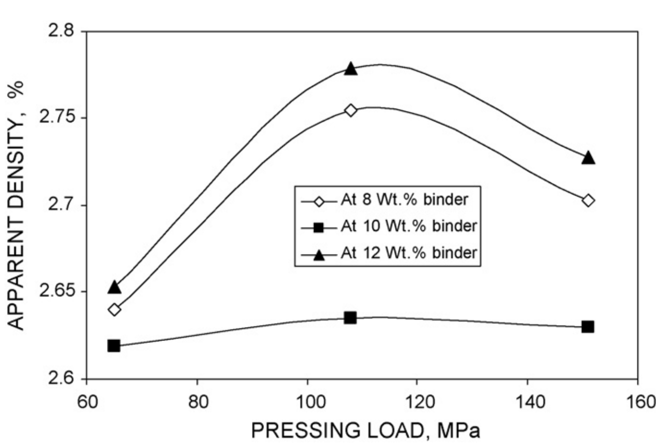

Fig. 5. Effect of pressing load on apparent density of green dolomite brick with different content of coal tar pitch binder.

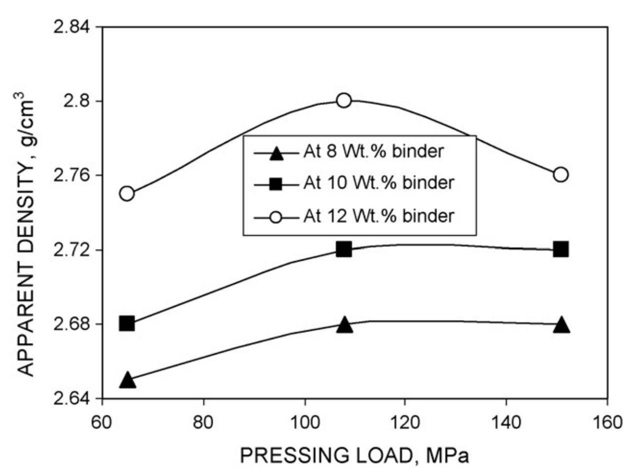

Fig. 7. Effect of pressing load on apparent density of calcined un-impregnated brick samples with different contents of coal tar pitch binder.

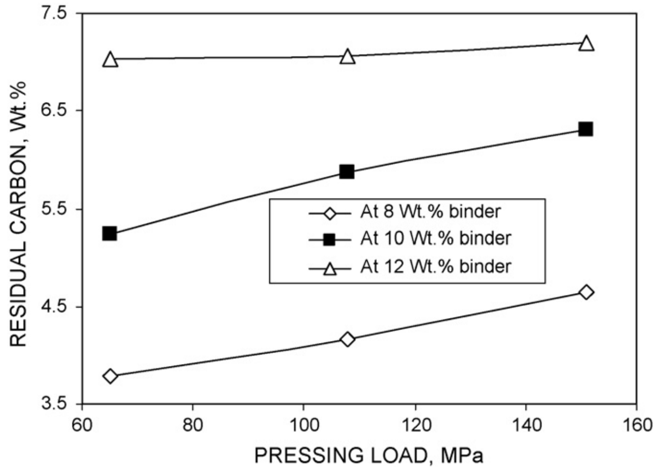

Fig. 8. Residual carbon of pitch-bonded dolomite after backing at temperatures up to 1000 °C as a function of pressing load.

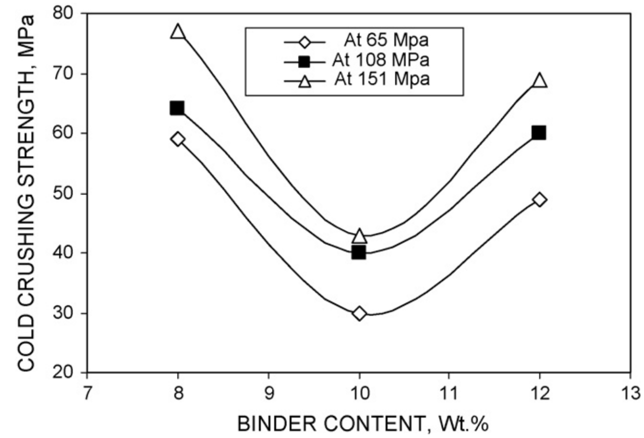

Fig. 9. Effect of the coal tar pitch binder on the cold crushing strength of green dolomite brick.

containing 8 wt.% binder exhibited hydration failure after 45 days. However, the dolomite bricks containing 10–12 wt.% binder failed after 52 days. Samples containing 10 wt.% binder and pressed under 108 MPa as well as those containing 12 wt.% binders and pressed at 151 MPa, show extraordinary hydration resistance (>60 days). Samples pressed at 65 MPa, whatever the content of binder, readily hydrated. This phenomenon can be explained due to the fact that such pressing load is not enough to seal the porous system. Hydration would take place if dolomite grain comes into direct contact with water molecules. Brick samples containing 10 wt.% binder and pressed at 108 MPa formed an impervious body in which dolomite grains are completely covered with a carbon film. Increasing the pressing load with the same binder content deforms the carbon film so that the dolomite grains are re-exposed to the water molecule. This effect can be compensated by increasing the binder content. Results given in Table 4 confirms these findings. This would suggest that hydration is a property dependent on the formed carbon framework in the brick body, which is a compromise between the binder content and the pressing load. The hydration resistance of samples containing 12 wt.% and pressed at 151 MPa are resistant to hydration because the formed carbon film is still established. However, pressing at lower load, implies that such load was not enough to attain the safe coating of the dolomite grains. The samples pressed at 108 MPa were selected for examining the effect of multi-impregnation.

Figs. 9 and 10 show the cold crushing strength (CCS) of the prepared samples as a function of the binder content. The green brick samples are stronger than to the baked bricks. In practice, for ladle furnace (LF) needs, the cold crushing strength of high performance green and baked carbon-bonded dolomite bricks amounts to 49 MPa and 44 MPa, respectively. The prepared samples acquired CCS values close to or higher than those of commercial quality bricks. The mechanical strengths of the bricks as a function of binder content shown in Figs. 9 and 10 can be explained by the filling in of their voids with a carbon framework. Voids is only satisfied with an adequate carbon framework with >9 wt.% binder. With lower binder content, the porous system of the brick body is not fully penetrated. Further increasing of the binder content up to 12 wt.% is enough to wet the dolomite contour and fill in the pores of the solid grains. Reasonably, the mechanical strength would attain its maximum. The mechanical strength of the baked samples behaves in a similar manner provided that the magnitude of the mechanical strength is lower than the green body. This is ascribed to the

Table 4
Hydration resistance of pitch-bonded dolomite

Pressed load (MPa)	Tar content (wt.%)	Lifetime in terms of hydration resistance
65	8	45 days
108	8	45 days
151	8	45 days
65	10	45 days
108	10	More than 60 days
151	10	52 days
65	12	52 days
108	12	52 days
151	12	More than 60 days
High performance carbon-bonded dolomite used in ladle furnace	–	30 days

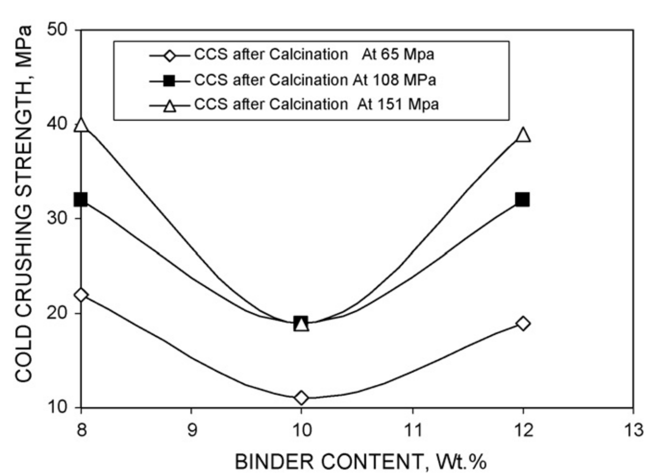

Fig. 10. Effect of the coal tar pitch binder on the cold crushing strength of the baked dolomite brick at temperatures up to 1000 °C.

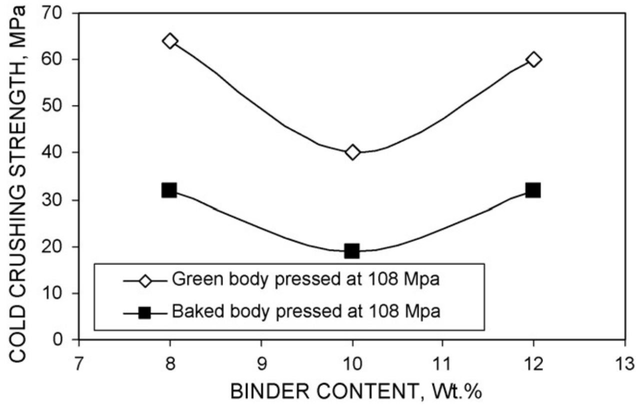

Fig. 11. Effect of the coal tar pitch binder on the cold crushing strength of dolomite brick.

thermal decomposition mechanism of coal tar pitch binder, initiation and propagation of the voids in the baked body. In Fig. 11, brick samples having 10 wt.% coal tar pitch binder showed the highest hydration resistance. As a result we now consider this formulation as the starting point for improving CCS.

Fig. 12 shows the effect of multiple impregnations on the density value of the dolomite brick samples having different binder content and pressing load. The density of the commercial brick amounts to 2.85 g/cm^3. Density increased gradually with number of impregnations attaining a constant value with the fourth impregnation. Density was 2.940 g/cm^3 after the fourth impregnation. Brick samples containing 12% pitch binder have higher densities than those containing less binder. This increase in density complies with the gradual decrease in the porous system of the fired brick with more impregnations. Density of the finished brick increased as the carbon filling in the voids to complete filling in the brick voids and porous system. Carbon also increased resistance of brick against water degradation. This assumption may be ascribed to the fact that the contact angle of water on carbon surface is high

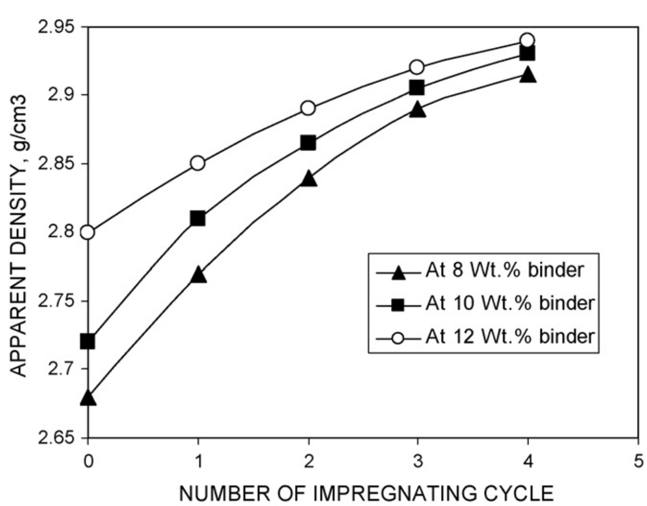

Fig. 12. Effect of the successive impregnating cycle on the apparent density of the baked coal tar pitch-bonded dolomite brick at temperatures up to 1000 °C and pressed at 108 MPa.

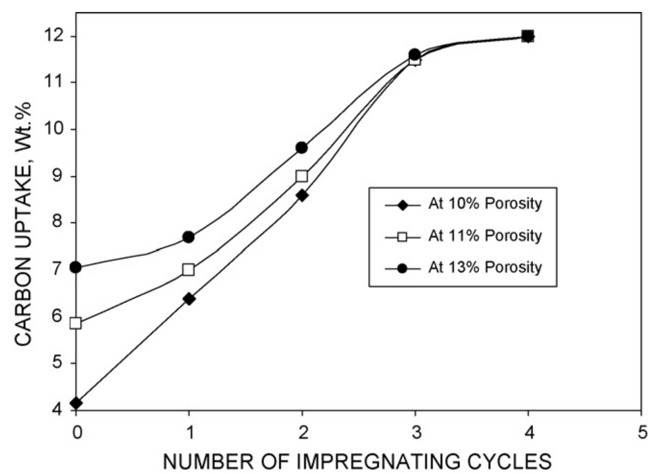

Fig. 13. Carbon uptake of the baked pitch-bonded dolomite brick pressed at 108 MPa as a function of number of impregnating cycles.

[22] compared to water/dolomite angle. In this work three multiple impregnations satisfied complete coverage of the dolomite grains with a continuous carbon film.

Fig. 13 shows the carbon uptake with the baked dolomite brick samples having different porosity values as a function of number of impregnation. It can be seen that the extent of carbon deposited in the pores of the brick samples increases gradually as the number of impregnations increased. The horizontal plateau of the curves given in Fig. 13 indicates complete filling of the pore system of the brick body with hard carbon. Multi-impregnation can thus be considered an essential process to produce dense bodies. The maximum extent of carbon uptake amounts to 12%. Fig. 14 shows the mechanical strength of baked dolomite bricks (10% binder) pressed at different loads as a function of carbon uptake taking place by multiple impregnations. The CCS increases with increase in the carbon uptake. High strength coal tar pitch-bonded dolomite bricks are attained with >11 wt.% carbon uptake and pressed samples ≥108 MPa. However, brick samples pressed under 65 MPa with 12 wt.% carbon uptake after impregnation also attained good CCS. Fig. 15 shows the cold crushing strength of the brick samples having different binder content and pressed under various loads. CCS values after 3 or

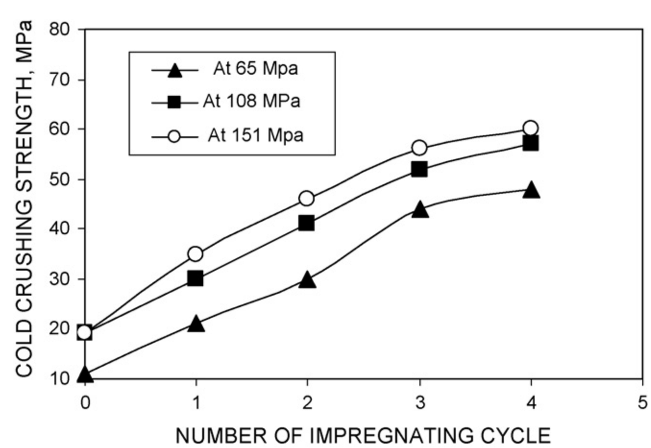

Fig. 14. Cold crushing strength of dolomite brick containing 10 wt.% of coal tar pitch as a function of carbon uptake/number of impregnating cycles.

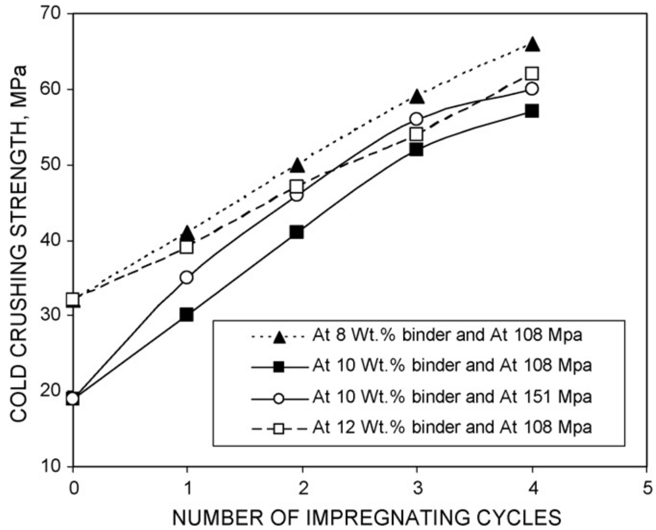

Fig. 15. Cold crushing strength of dolomite brick containing 8–12 wt.% of coal tar pitch at different pressing load as a function of carbon uptake/number of impregnating cycles.

more impregnations are $\geq$49 MPa in bricks containing 8–12 wt.% binder, pressed under 108–151 MPa and >11 wt.% carbon uptake, so that a high quality brick is obtained compared to the commercial market quality brick (CCS ~44 MPa).

4. Conclusion

In this study, successful preparation of multi-impregnated pitch-bonded Egyptian dolomite refractory brick having outstanding properties meeting the requirements of Ladle furnace (LF) for steel making industry was prepared. Dolomite bricks containing 8–12 wt.% coal tar pitch binder and pressed under 108–151 MPa acquired high cold crushing strength. Multi-impregnating favored the mechanical strength of the baked brick samples and improved their hydration resistance (>45 days). Samples are found high quality compared to the conventional and commercial quality bricks. However, dolomite brick containing 10 wt.% coal tar pitch and pressed at 108 MPa gave high hydration resistance (more than 60 days in normal condition) compared to the hydration resistance of the commercial quality bricks (30 days).

The finished brick withstand humidity degradation effect during storage for periods up to >60 days. Estimation of production cost of the brick indicates it is competitive with the market price based on durability and service life time aspects.

5. Preliminary cost study

Cost estimation of the finished brick was carried out on terms of durability, service life time and stability against hydration attack during storage. Results will be published elsewhere provided that the cost study shows that the price of the finished brick highly competes the price of local market for high quality dolomite brick.

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