

PREPARATION OF PRECIPITATED CALCIUM CARBONATE FROM WADI GHADAF LIMESTONE OF DAMMAM FORMATION

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ABSTRACT

In view of the availability of a cheap and a high quality limestone raw material and the practical abilities to produce precipitated calcium carbonate (PCC) using the carbonation process route, this study was carried out with bench scale experiments. High purity limestone of Wadi Ghadaf (Dammam Formation), was calcined to produce quick lime which was converted to calcium hydroxide by slaking with water, the final stage comprised injection of CO₂ gas into the slaked lime slurry. Calcium carbonate was precipitated as a high purity (99.5% CaCO₃) white powder (95% brightness) with a particle size range of (6 – 12) microns. Conditions optimization was done through each stage of this study.

تحضير كربونات الكالسيوم المترسبه من حجر الكلس لمنطقة وادي غداف

وسن عبد الكاظم مسلم و علاء محمد خليل مصطفى

المستخلص

على ضوء توفر المادة الأولية المتمثلة بحجر الكلس بنقاوة جيدة وبسعر رخيص مع وجود الإمكانية العملية لتطبيق طريقة الكربنة، تم إجراء تجارب منضدية لتحضير كربونات الكالسيوم المترسبه حيث تم أولاً حرق حجر الكلس من منطقة وادي غداف (تكوين الدمام) لإنتاج النورة الحية (أكسيد الكالسيوم) التي تمت معاملتها بعد ذلك بالماء لإنتاج هيدروكسيد الكالسيوم وتبع ذلك ترسيب كربونات الكالسيوم بحقن غاز ثاني أكسيد الكربون في لباب الهيدروكسيد، تم تثبيت الظروف المثلى لكل مرحلة من مراحل تحضير كربونات الكالسيوم المترسبة والتي أعطت نتائج جيدة للمنتج من خلال نعومة تراوحت بين (6 - 12 مايكرون) ونقاوة عالية حوالي (99.5%) ودرجة بياض 95%.

INTRODUCTION

Limestone (mainly CaCO₃), is an abundant rock found in all parts of Iraq and the world, and is the main component of shells of marine organisms, snails, pearls, and eggshells. Industrial calcium carbonate is produced in two ways; by extracting and grinding the natural ore (ground calcium carbonate GCC) and by chemical precipitation (precipitated calcium carbonate PCC). Ground calcite in the form of chalk, and limestone is perhaps the most widely available and utilized natural mineral in the world additives, available in a wide range of particle sizes, which function as low cost fillers. In comparison synthetic PCC with a very fine and controlled particle size offers a range of technical effects beyond the capability of (GCC) fillers and other more expensive and sophisticated additives used to improve, the physical characteristics of finished products.

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The usage of PCC as a white pigment offers a wide variety of benefits in terms of gloss, brightness, opacity and viscosity. PCC is considered as one of the most versatile chemicals used in the modern paint, coatings, rubber, plastics, glass, textiles, putties, chalks, sealant, adhesives, varnishes, food, cosmetics, dentifrices, chemicals and pharmaceuticals, ink and paper making markets (Jasra *et al.*, 2004). In North America, calcium carbonate has begun to replace kaolin in the production of glossy paper. Europe has been practicing this as alkaline papermaking or acid-free papermaking for some decades.

Precipitated calcium carbonate is commonly manufactured by the reaction of gaseous carbon dioxide with a colloidal suspension of calcium hydroxide which is sufficiently versatile to allow specific crystal morphologies to be developed.

In Iraq there is no such industry for production of PCC, despite the availability of high purity limestone (such as that of the Dammam Formation in Wadi Ghadaf, Western Desert of Iraq), besides the wide need of PCC in several local industries, as a filler, extender, and coating pigment. The aim of this study is to evaluate the preparation process of precipitated calcium carbonate from Wadi Ghadaf limestone, and establish the process route by bench scale tests.

▪ Previous Work

PCCs have been manufactured commercially for more than a century. The first producer was the English John E. Sturge Ltd. company, which treated the residual calcium chloride from their potassium chlorate manufacture with soda ash and carbon dioxide to form what they called precipitated chalk. Since 1920, chemically precipitated calcium carbonate has been used as a pigment or filler in the paper industry (Bleakley and Johns, 1994). Various chemical routes, however, have been followed to precipitate the calcium carbonate (Teir *et al.*, 2005) but the most frequently used methods were based on the double decomposition of sodium carbonate with either calcium hydroxide or calcium chloride. The first one is called the Lime Soda Process, where calcium hydroxide is reacted with sodium carbonate to produce sodium hydroxide solution, from which the calcium carbonate is precipitated. In this process PCC is produced as a by-product, while sodium hydroxide is the main product. The second method is the Solvay process, whereby calcium hydroxide is reacted with ammonium chloride, forming ammonia gas and calcium chloride solution. After purification, this solution is reacted with sodium carbonate to precipitate calcium carbonate. This process is the simplest one, but economically depends on the availability of low cost calcium chloride. PCC was also produced from the reaction of calcium hydroxide with bicarbonate solution which is generated from water softening process. However, the most widely used process of carbonation was carried out with carbon dioxide gas injection in an aqueous suspension of calcium hydroxide "milk of lime", due to the cheap raw materials used (Bleakley and Johns, 1994).

The most applied process of PCC production is based on the carbonation of "milk of lime". It is performed in three stages; firstly, the calcinations of raw limestone to produce calcium oxide or "quicklime"; secondly, the "slaking" of the quicklime with water to produce an aqueous suspension of calcium hydroxide; and finally, the carbonation of the calcium hydroxide with a gas comprising carbon dioxide.

Hall and Wyandotte (1960) were concerned with the production of precipitated calcium carbonate by various chemical routes for the paper industry. A process based upon the carbonation of "milk of lime" is preferred because there is no serious problem of

contamination of the product with unwanted salts, and each of the three stages in the production process can be controlled to adjust the properties of the final product.

Since 1992, almost all PCC is made by direct carbonation of hydrated lime, known as the Milk of Lime Process. A revolution in wood-free paper making has occurred over the past decade; also it is well known that acid papers degrade over time. The poor archival properties of acid wood-containing papers diminish their value. The availability of low-cost precipitated calcium carbonate (PCC), manufactured on individual paper mill sites, and fueled the rapid expansion of alkaline paper making. PCC provides many benefits, including excellent brightness and opacity at significantly lower cost than the clay or fiber it replaces (Ain and Laleg, 1996).

An environmentally friendly process was described by Jasra *et al.* (2004) for the production of precipitated calcium carbonate suitable for industrial application from a calcium carbonate-rich by-product of a nitro phosphate fertilizer plant. The process employs purification steps of calcination, carbonation and optional treatment with an emulsion of fatty acid or fatty acid derivatives.

Bleakley and Johns (1996) described a method of preparing precipitated calcium carbonate which comprises: **1)** slaking quick lime in an aqueous medium, **2)** subjecting the aqueous medium to continuous agitation during the said slaking, **3)** passing the suspension of calcium hydroxide obtained after slaking through a sieve having an aperture size of 40 – 70 microns, **4)** subjecting the suspension to high shear agitation with an impeller having a peripheral speed of 40 – 70 m/sec., so as to obtain finely dispersed calcium hydroxide, **5)** terminating the said high energy high shear agitation on achieving finely dispersed slaked lime, **6)** carbonating the finely dispersed slaked lime by passing sufficient carbon dioxide gas to neutralize the pH of the suspension during this carbonation step, **7)** subjecting the suspension to continuous agitation with an impeller speed of 200 – 700 cm/sec to maintain the suspension, and **8)** separating the precipitated calcium carbonate formed in the process. The disadvantage associated with this method is the requirement of generating high energy high shear agitation during slaking and carbonation. Furthermore, Hammodi and his colleagues (Hammodi *et al.*, 1998) evaluated the preparation of precipitated calcium carbonate from hydrated lime which was produced as byproduct from The Iraqi sugar production firm in Mosul.

MATERIALS AND METHODS

▪ Materials

— **Limestone:** Wadi Ghadaf limestone deposits belong to the Dammam Formation, Al-Faj-unit; Middle Eocene (Upper Lutetian) is located in the Western Desert of Iraq, about 100 Km southwest of Ramadi and 75 Km from Baghdad-Rutba highway (Seryoka, 1986). These deposits were specified previously as the productive bed of limestone that met the requirements for white cement and glass industries, with industrial bed thickness of about (11 m). The limestone deposit was divided into two parts the 1st for white cement industry and the 2nd (which is more pure) for glass industry with a reserve estimated by about 38 million tons.

The quality of limestone depends on its purity especially the presence of colored impurities such as iron oxide (the main factor), manganese oxide, and insoluble materials. The chemical composition as well as XRD pattern of Wadi Ghadaf limestone samples, which were obtained from that supplied for the glass industry, is shown in Fig. (1) and (Table 1).

Table 1: Chemical composition of raw limestone of Wadi Ghadaf

Composition	CaO	Fe ₂ O ₃	MgO	SO ₃	Al ₂ O ₃	P ₂ O ₅	IR	L.O.I
%	54.78	0.089	0.15	0.07	0.13	0.04	0.95	43.12

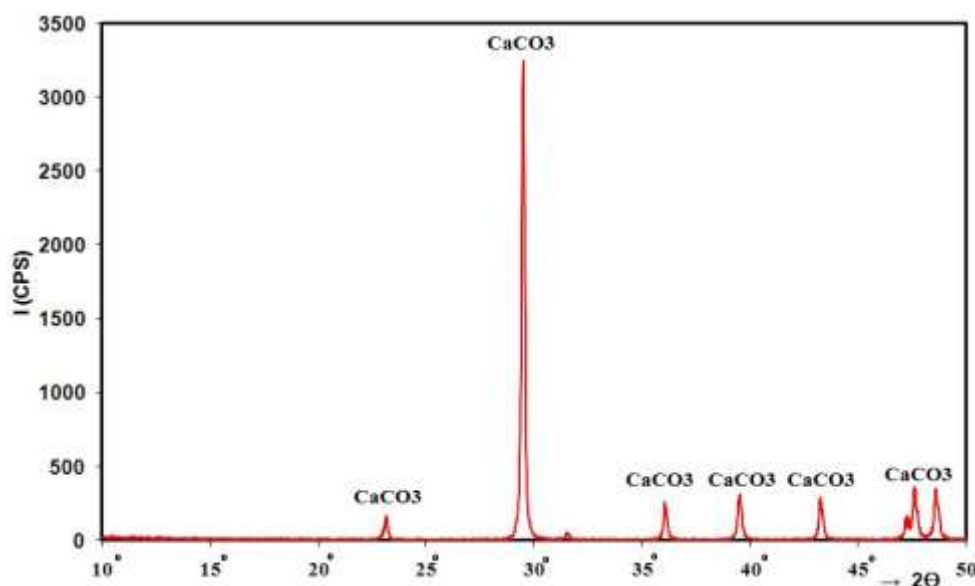
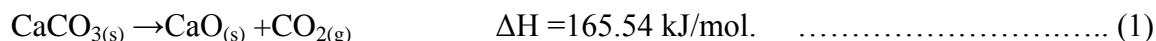


Fig.1: XRD pattern of Wadi Ghadaf limestone sample

▪ Methods

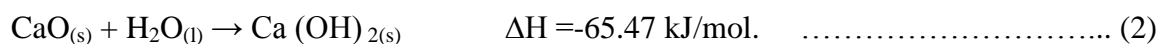
The process performed in this work is comprised of three stages:

— **Calcinations of limestone:** Crushed limestone with particle size range of (4 – 10, 2 – 4, and 1 – 2) cm and a ground sample of (75μ) is burned in a muffle furnace, for decomposition into calcium oxide (quicklime) and carbon dioxide:



Calcination was carried out at different temperatures (800, 900, 1000, 1100, and 1200) °C for variable residence time (30, 60, 90 and 120) minutes and the heating rate was set at (10°C/min.). At the end of the calcination, weight of the cooled quicklime samples was taken immediately to determine the loss in weight for each test.

— **Slaking of lime:** The quicklime produced from calcination was slaked with excess water using different solid (15, 20 and 25) wt.%. The slurry was stirred vigorously for different time periods (15, 30 and 60) minutes. Reaction can be represented according to the following equation:



The slurry (milk of lime) was screened through (45μ) sieve to remove impurities originated from the limestone. Samples were drawn for chemical and XRD analysis to determine the calcium hydroxide (milk of lime) content.

— **Carbonation of slaked lime:** The milk of lime was carbonated by bubbling carbon dioxide gas into the slurry to precipitate CaCO₃ according to the hydrothermal reaction:



The precipitation of calcium carbonate, however, was studied at various reaction times (15, 30, and 60 min.), carbonation temperatures (10, 35, 50, and 90) °C, solid % (10, 15, and 20 wt %) and different CO₂ flow rates (708, 424.8, 283.2, 212.4, and 141.6 lit/h). The purity of the precipitated calcium carbonate samples were tested by chemical analyses and X-Ray diffraction to get the optimum conditions of carbonation process. Industrially, CO₂ gas is recycled from the calcination unit after washing with water to reduce cost.

RESULTS AND DISCUSSION

▪ Calcination of Limestone

As it has been stated above, the production of precipitated calcium carbonate (PCC) is a multi-stage process. Calcination of the limestone is the initial and essential stage in the process; therefore, several parameters were investigated through this stage to determine the best calcination conditions. The parameters that were studied are as follows:

— **Effect of calcination temperature:** The usual theoretical temperature used in traditional lime kiln process is around 900 °C (Moropoulou *et al.*, 2001), but in practice to evaluate the dissociation completion of limestone to quick lime, samples of limestone were calcined at different temperatures (800, 850, 900, 950, 1000, 1100 and 1200) °C. To determine the correct temperature in the kiln is an art rather than a science (Hassibi, 2009), and the weight loss of the samples after calcination corresponds to the released amount of CO₂ gas during the process. The weight loss is plotted as a function of the calcination temperature Fig. (2).

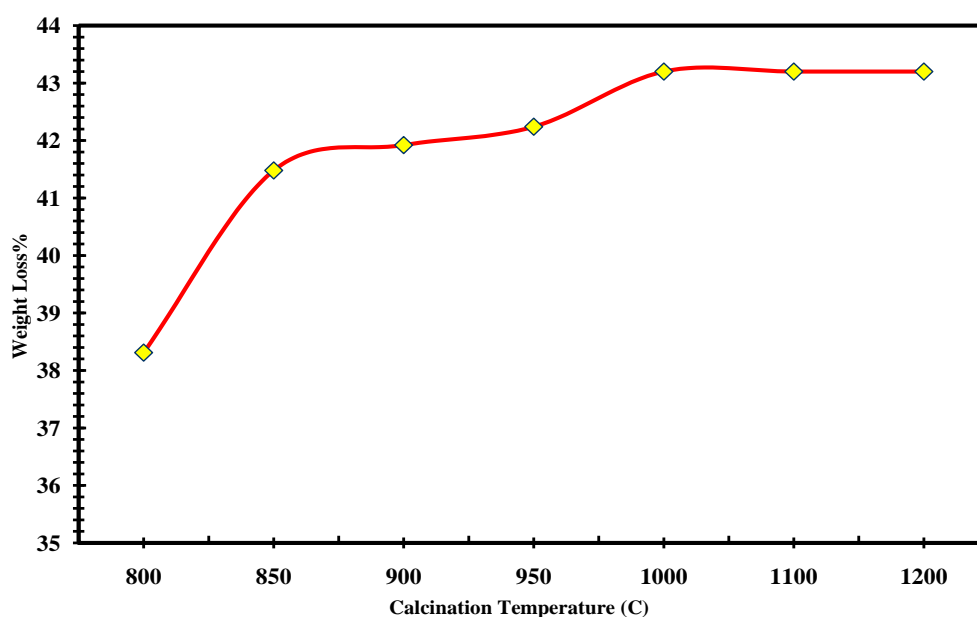


Fig.2: Effect of calcination temperature on the weight lost

According to the chemical equation (1) given above, the theoretical percentage of weight loss due to the dissociation of limestone into quick lime (CaO) and carbon dioxide (CO₂) gas is 44%. As shown in Fig. (2), the higher the calcination temperature the higher the loss in weight which means that more conversion occurs, but it can be noticed that after 1000 °C, there is no significant increase in the weight loss with low reactivity of the produced quicklime because higher temperature will increase shrinkage (hard burned lime) (Hassibi, 2009). Therefore, a temperature of 1000 °C can be considered as an optimum temperature for Wadi Ghadaf limestone calcination.

— **Effect of calcination (soaking) time:** Soaking time is another factor that could affect calcination efficiency. Accordingly, it takes time for heat penetration. If the soaking time is too short, the core of the limestone will remain calcium carbonate, while the outside will convert to calcium oxide. If the soaking time is too long, the surface of the pebbles will shrink and the pores created by generating of CO_2 gas will close, producing an impervious surface. This leads to what is called “hard burned” or “dead burned” lime, which has a low chemical reactivity. In addition, longer soaking time means low production rate and higher costs of production. For these reasons, several experiments were carried out using different calcination times (30, 60, 90 and 120) minutes, at 1000°C . The results are shown in Fig. (3).

It is clearly seen from Fig. (3) that 98.2% of the theoretical loss in weight due to the CO_2 emissions occurred during the first 60 minutes of calcination. Retention time at 30 minutes have the lower weight loss percentage because the core of the limestone pebbles will remain calcium carbonate while the outside will convert to calcium oxide. Longer retention time (90 and 120) min. means less production and higher costs of manufacturing (Hassibi, 2009).

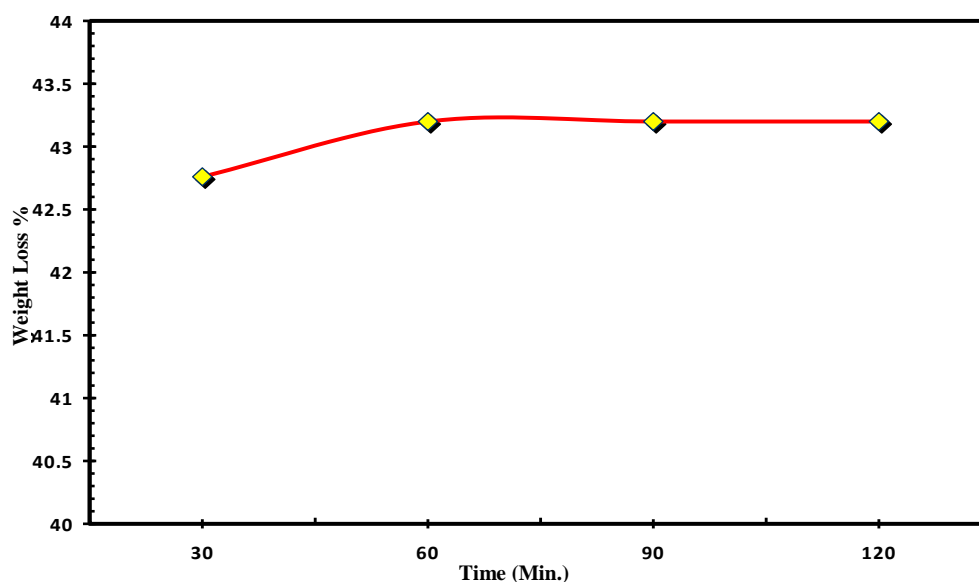


Fig.3: Effect of calcination time on the production of quick lime

— **Effect of limestone feed particle size:** The particle size distribution must be compatible with the requirements of the kiln. The experiments are driven with muffle furnace, which is different from the two practical major types (shaft and rotary) kilns used in calcination of limestone, and must be fed with a range of limestone particle size (1 – 25) cm and (dust – 6 cm). Thus, a suitable range of the feed particle size, depends on the kiln type. The present experiment is carried out with the whole particle size range of the two types of kilns (4 – 10, 2 – 4, and 1 – 2) cm and a ground size of ($-75\mu\text{m}$).

It is obvious from Fig. (4) that the limestone feed with a range of (4 – 10) cm particle size resulted in a higher weight loss during calcination at 1000°C . This may be attributed to the rehydration occurring with the reduction in particle size. Another reason to prefer 4 – 10 cm particle size over the other particle sizes of limestone is the slow heat evolved in the next stage of slaking, so as to produce more homogeneous slaked lime. So that the vertical shaft kiln is suitable with this pebble particle size.

XRD pattern is shown in Fig. (5) of the CaO produced from calcining limestone sample of particle size 4 – 10 cm at 1000 °C for one hour which is taken as the optimum condition of limestone calcination.

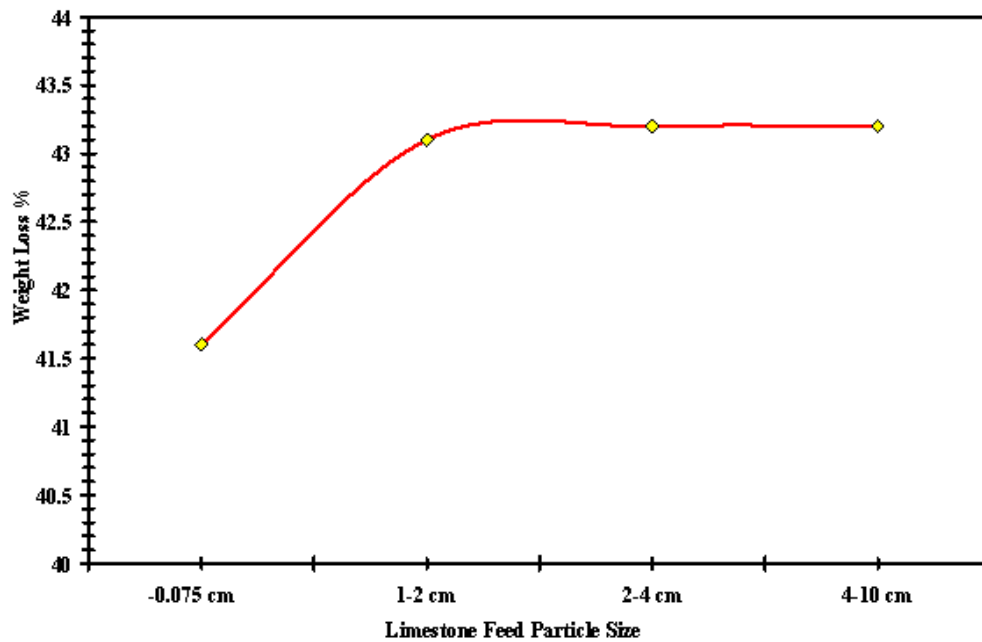


Fig.4: Effect of limestone feed particle size on the production of quick lime

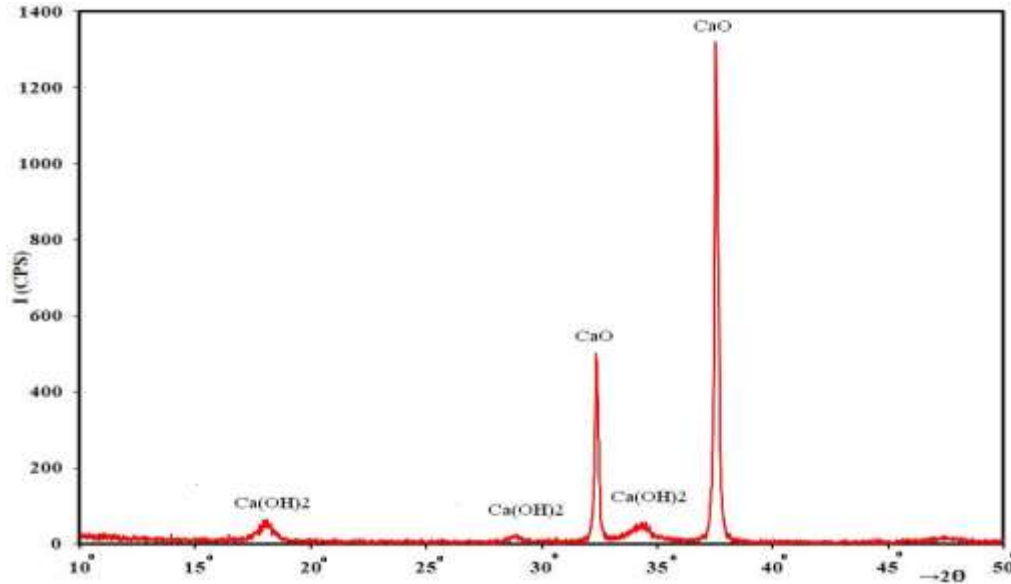


Fig.5: XRD pattern of quick lime produced at optimum conditions

▪ Slaking of Lime

Technically, hydration processes typically yield a dry powdered hydrate, whereas slaking involves more water, producing wet hydrates (Boynton, 1980). During hydration of quicklime two simultaneous phenomena were observed: temperature increases due to the exothermic reaction and expansion of the volume of the paste. The mode of temperature during the slaking time is illustrated in Fig. (6) as temperature versus time.

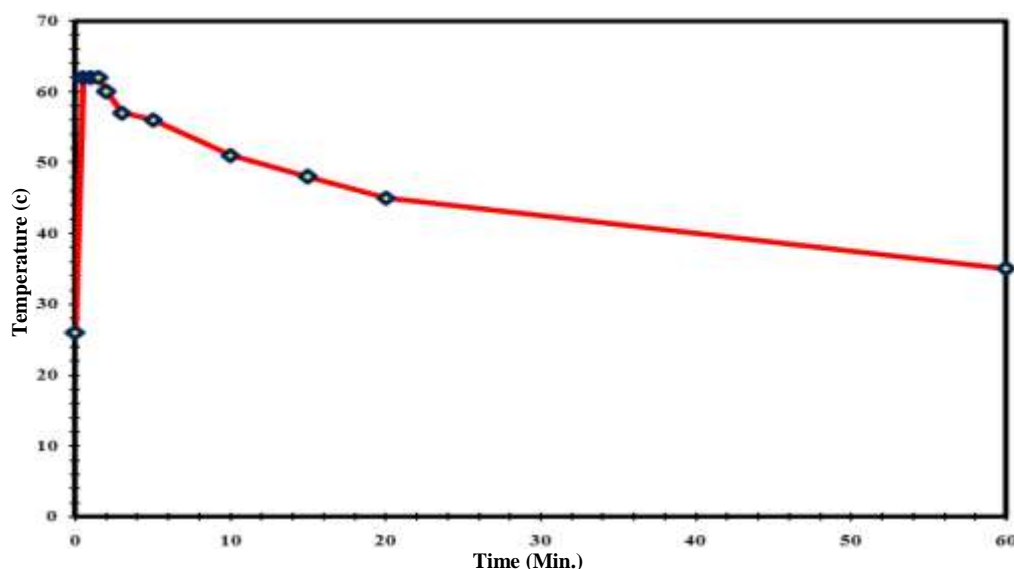


Fig.6: The rate of temperature versus slaking time

The rate of slaking and optimum hydration conditions must be empirically and individually established. In this stage quicklime from the previous calcination process was slaked with sufficient amount of tap water which is suitable for slaking because it should contain sulfates or sulfites of no more than (500) mg/L (Boynton, 1980). Several parameters such as (solid%, time, particle shape and size) were studied and their effects were evaluated.

— **Effect of solid weight percentage:** The main distinguishing characteristic of the hydrated (hydroxide) forms of lime is the amount of excess water they contain, which does not include dilution or process water that may be added to slaked lime downstream of the slaking compartment. These forms are discussed below (Boynton, 1980):

- Dry Hydrate: The most concentrated form of hydrate, a dry, fine, white powder. The commercial products usually contain 72 – 74 % lime (CaO) solid.
- Putty: It is a wet, amorphous-appearing, plastic paste form of hydrate that has “body,” so that it is moldable, a thixotropic mass. The oxides or lime solids contents range between 40 – 60 %.
- Slurry: It is a colloidal suspension form of hydrate which has the consistency of a thick cream. Unlike putty, it has no body or plasticity, and behaves like a viscous liquid. It contains about 25 – 35 % of lime solid content.
- Milk of lime: It has the consistency of whole milk, which is considerably thinner than slurry, and flows almost as readily as water. It has a lime solids content ranging from 1 to 20%.
- Lime water: It is a pure, saturated or unsaturated aqueous solution of calcium hydroxide without any lime solids.

According to the highlighted types of hydrated lime forms shown above, four lime solid percentages (10, 15, 20 and 25) were used to obtain the best solid % for slaking, which was identified through Ca(OH)_2 % of the products.

As it is shown in Fig. (7), the Ca(OH)_2 percentage increases as solid percentage increases from 10% to 15% then slightly increases to 20% and clearly decrease with the increasing in lime solid percentage. This relationship is maintained up to 20%. However, the Ca(OH)_2 percentage shows a sharp decrease when lime solid increases from 20% to 25%. Taking into consideration the economic factors, 20% of solid lime seems to be reasonable. When the amount of slaking water increases the rate of hydration is retarded and mutes the heat evolved in the diluted mixture which retards the slaking and incomplete hydration occurs. Also adding insufficient water results in an incomplete hydration of the lime (Boynton, 1980).

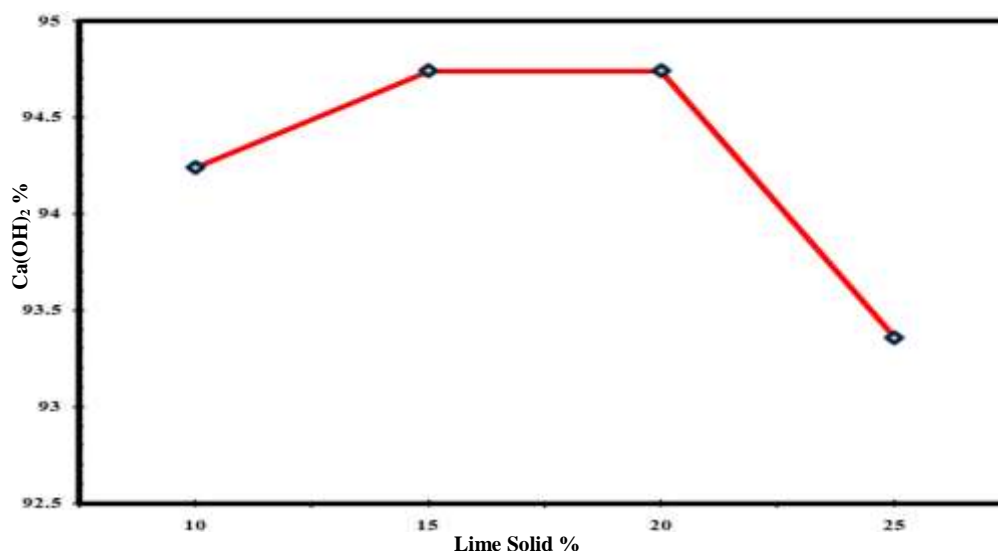


Fig.7: Effect of lime solid percentage on the production of calcium hydroxide.

— **Effect of slaking time:** Slaking time is the time required to complete hydration. This time varies from lime to lime. In literature, various times were mentioned to be sufficient for the complete slaking (hydration reaction). From the results shown in the Fig. (8), it can be concluded that 15 minutes of slaking time is an optimal time and beyond it almost no increase in Ca(OH)_2 occurs. At this time (15 minutes) a conversion of Ca(OH)_2 of about 90.7% was obtained. This agrees with many previous studies which refer that slurry slakers are designed for a retention time of (10) minutes at a full rated capacity (Hassibi, 2009; Vanderheiden and Easton, 1983; Bleakley and Johns, 1996). Hence, different periods of time were applied to detect the best slaking (hydration) time. The hydrator typically provides an average residence time of 10 to 15 min.

The XRD pattern of Ca(OH)_2 sample produced at the optimum conditions (lime solid 20% and 15 minutes of slaking time) is shown in Fig. (9).

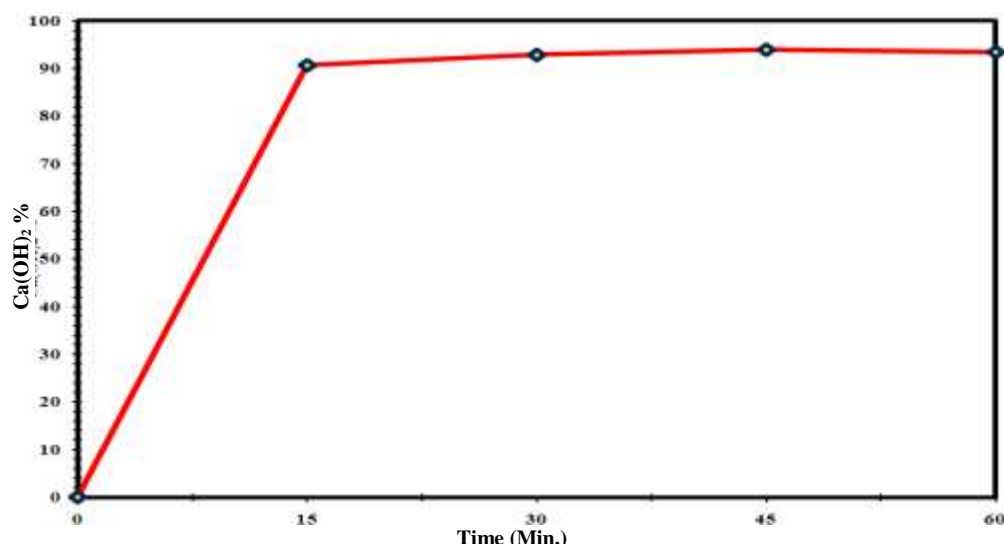


Fig.8: Effect of slaking time on the production of calcium hydroxide

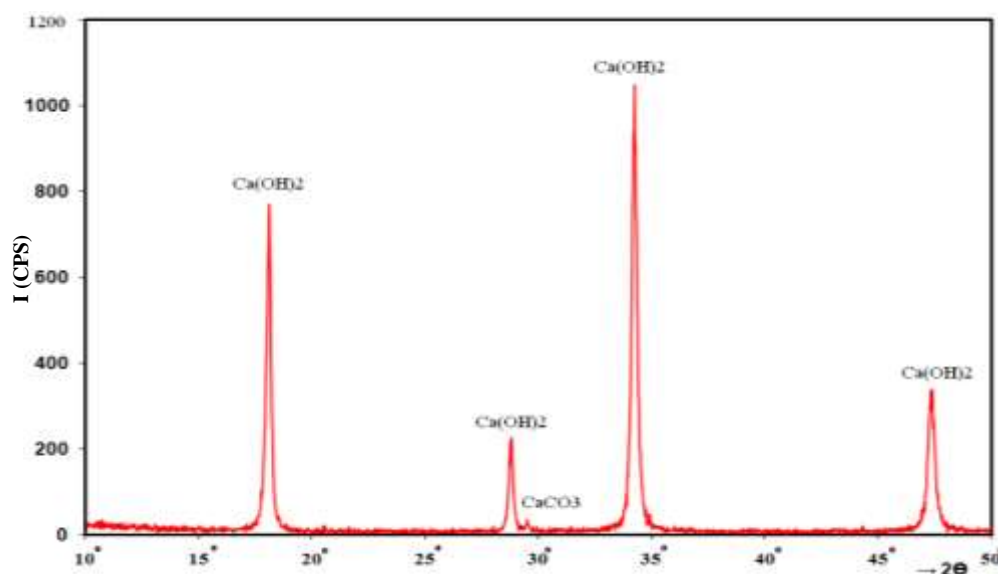


Fig.9: XRD pattern of Ca(OH)₂ produced at optimum conditions

▪ Carbonation of Slaked Lime

Virtually all commercial PCC is produced by reacting milk of lime with carbon dioxide. The process is complex and involves simultaneous dissolution of calcium hydroxide and carbon dioxide, and crystallization of calcium carbonate. Carbonation is generally carried out in a series of reactors under closely controlled pH, temperature and degree of supersaturation, to produce the required PCC. Crystallization can occur on the surface of the calcium hydroxide particles (producing scalenohedral crystals), in the aqueous phase (producing rhombohedral crystals) and at the gas-liquid interface (Oates, 1998). In this work several factors have been studied to optimize the production of PCC. These factors include the following:

— **Effect of carbonation time:** It can be defined as the period of time when calcium hydroxide suspension or slurry is in contact with carbon dioxide gas. With proceeding of reaction time, the PCC is simultaneously produced, which reduces the amount of calcium

hydroxide remain in the slurry. Richard and Wood (1977) injected CO_2 to the slurry of Ca(OH)_2 in two stages over a total time of about 70 – 90 minutes. Vanderheiden and Easton (1983) carried out one stage carbonation process over a period of about 37 minutes. Jasra *et al.* (2004) followed another approach to determine sufficient time of carbonation. They allowed carbon dioxide passing through the slurry until pH of the slurry falls to near neutral.

In this study, carbon dioxide was allowed to pass through slurry of 15 wt.% calcium hydroxide for periods of (15, 30 and 60) minutes. From Fig. (10) it can be easily noticed that carbonation time has significant effect on the PCC%. Hence, at 60 minutes a remarkable PCC productivity of 99.73% was achieved.

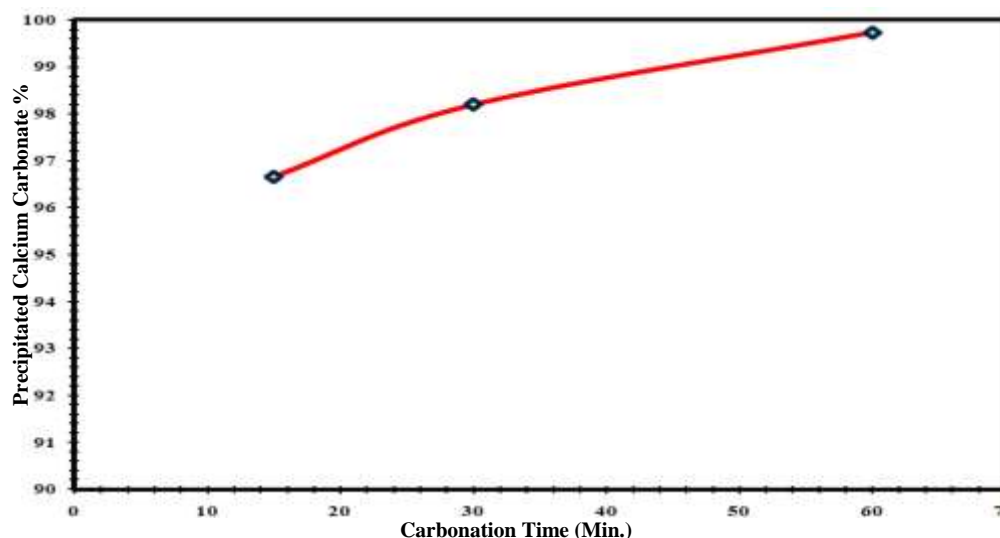


Fig.10: Effect of carbonation time on the PCC%

— **Effect of carbonation temperature:** To study the effect of temperature of the calcium hydroxide slurry during carbonation, four experiments were conducted at different temperatures (10, 35, 50 and 90) °C at a constant flow rate of 708 (lit/hr)/kg of Ca(OH)_2 to evaluate the best temperature of carbonation. The dried products of experiments were investigated by microscope to determine the particle size distribution as shown in Table (2).

Previous studies discuss the effect of temperature on the carbonation of slaked lime. Bleakley and Johns (1996) and Teir *et al.* (2005) mentioned that calcium carbonate can be precipitated from aqueous solution in three different crystal forms: the vaterite form which is thermodynamically unstable, the calcite form which is the most stable and the most abundant in nature and the aragonite form which is metastable under normal ambient conditions of temperature and pressure. Aragonite converts to calcite at elevated temperature where the threshold temperature for the aragonite structure is approximately 49 °C.

Calcite form exists in several different shapes of which the most commonly found are the rhombohedral shape in which the length and the diameter of the crystals are approximately equal at a carbonation temperature in the range (– 1 – 18) °C, and the scalenohedral shape, in which the crystal are like double, two-pointed pyramids, can be formed in carbonation temperature range of (49 – 90) °C which agrees with the results of the PCC particle size and structure observed under the microscope, as shown in Table (2).

Table 2: PCC product particle size and structure at deferent carbonation temperatures

Carbonation Temp. °C	10	35	50	90
Dominant Particle Structure	sub rounded	sub rounded	sub rounded subhedral	sub rounded subhedral
Dominant Particle Size (micron)	6 12	12	12 24	12 24

The conclusion extracted from the Fig. (11) is that the best PCC% (about 99.73) % can be prepared under a range of temperatures (30 – 40) °C. Specifically 35 °C is the best temperature for carbonation according to Table (3) that produced PCC with homogeneous dominant particle size of about (12 µm). While at (10) °C the particles is finer (down to 6 µm) which is more preferable.

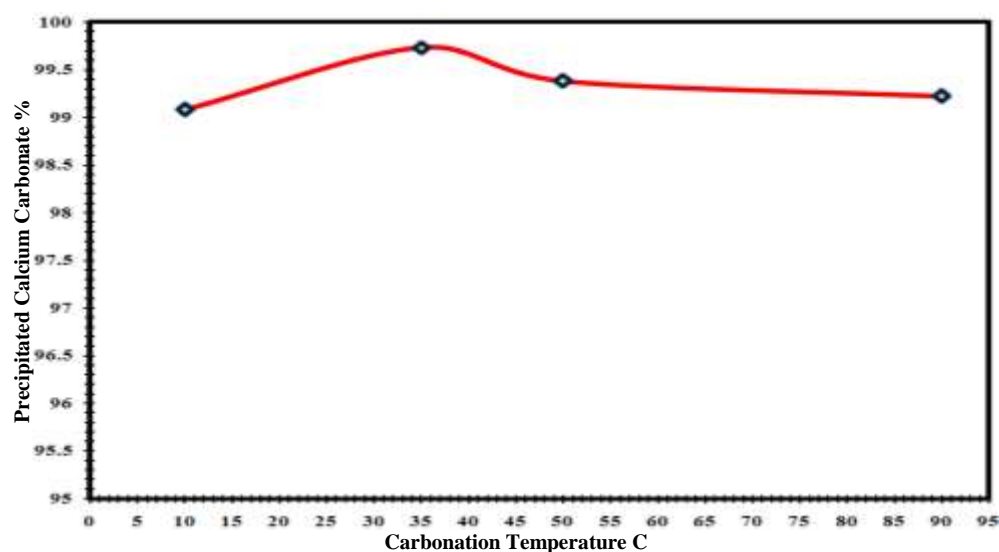


Fig.11: Effect of carbonation temperature on the PCC%

— **Effect of slaked lime solid wt. percentage:** According to known literature, wide ranges of hydrated lime solid percentage have been applied for optimum carbonation results. However, a minimum of 0.1 and a maximum of 20 solid percentage were used (Richard and Wood, 1977; Shibazaki *et al.*, 1979; Vanderheiden and Easton, 1983; Bleakley and Johns, 1996; Jasra *et al.*, 2004). In this work, several weight percentages of hydrated lime were used (10, 15 and 20) %.

As shown in Fig. (12), the results indicate that when hydrated lime solid percentage increases beyond 15%, PCC percentage decreases. Mathur (2001) mentioned that carbonation reaction is an equilibrium reaction, more dissolution of the calcium hydroxide takes place from the lime slurry to increase the concentration of calcium ion up to solvent solubility limits (inverse temperature dependence phenomena), until all of available calcium hydroxide is dissolved, and all the available calcium ions are converted to calcium carbonate. Meanwhile, very low solid percentage drives the whole process to be uneconomic. Therefore, calcium hydroxide is preferably prepared at (15%) solid percentage.

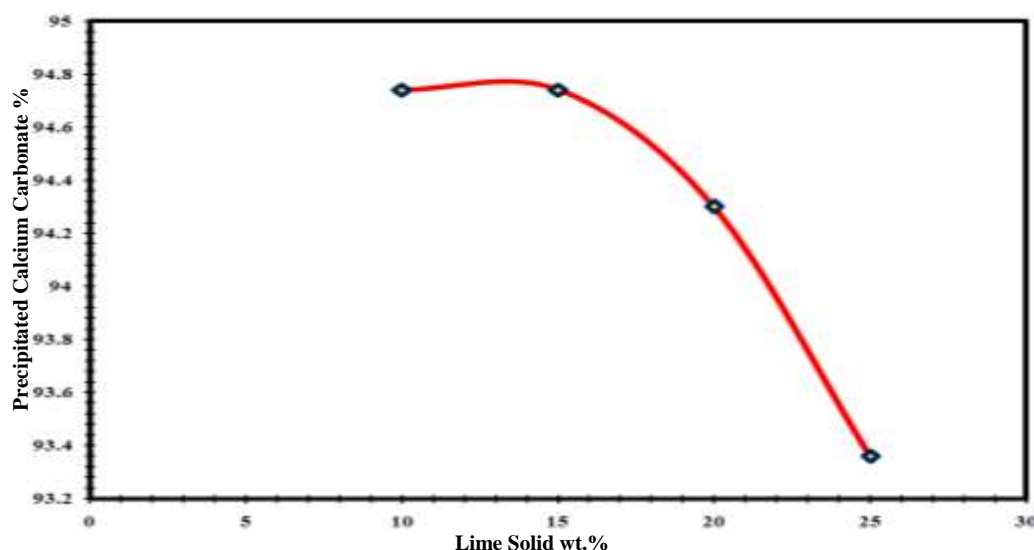


Fig.12: Effect of slaked lime solid wt.% on the PCC%

— **Effect of CO₂ gas flow rate:** Flow rate is an essential factor affecting the carbonation process. Hydrated lime should receive sufficient quantity of CO₂ gas to convert into PCC. The lesser quantity of CO₂ gas, the lower the amount of PCC produced and vice versa. Figure (13), shows the relation between CO₂ flow rate, and the resulted PCC wt.%.

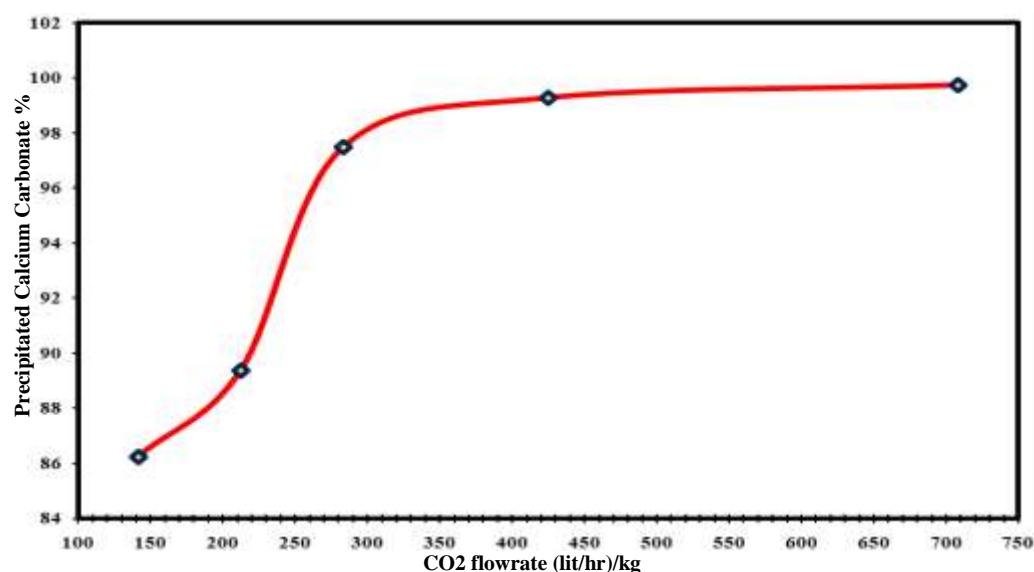


Fig.13: Effect of flow rate of CO₂ gas on the PCC wt.%

Five values of flow rate (141, 212, 283, 424 and 708) (lit/hr)/kg of hydrated lime, were applied to optimize the PCC percentage production. As it can be seen from Fig. (13), the PCC productivity (wt.%) highly increases as the CO₂ flow rate increases to about 283 (lit/hr)/kg and significantly remains constant as this value is exceeded. A considerable high enough productivity of PPC 99.27% produced using a flow rate of CO₂ gas of about 425 (lit/hr)/kg. As it is shown in Fig. (3), every increase in CO₂ gas flow rate is reflected by an increase in PCC percentage. Other workers Vanderheiden and Easton (1983), and Bleakley and Johns (1995), used a flow rate of 685 and 846 (lit/hr)/kg respectively, which are much higher than our findings.

Precipitated calcium carbonate (PCC), produced at optimum conditions, CO₂ gas flow rate 425 (lit/hr)/kg of calcium hydroxide, carbonation temperature 10 °C, carbonation time 60 minutes and a solid percentage of about 15%. The chemical composition of the product is shown in Table (3). However, brightness of the product sample is measured by the Color Flex spectrophotometer model no. (45°/0°) gave about (95%). The XRD pattern of the product is shown in Fig. (14) below.

Table 3: the chemical composition of the precipitated calcium carbonate produced at optimum conditions

Composition	CaO	Fe ₂ O ₃	MgO	IR	L.O.I
%	55.55	0.02	0.18	0.29	43.88

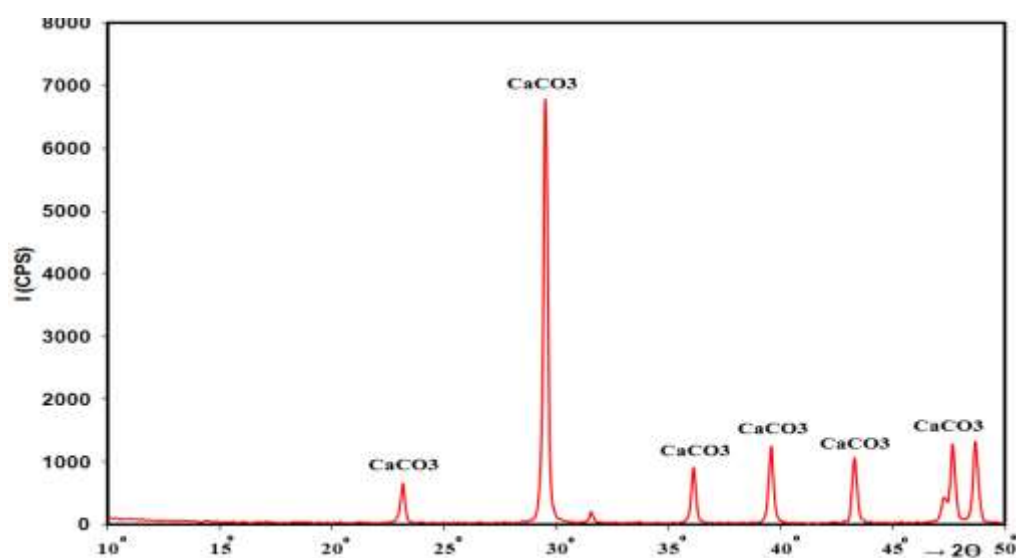


Fig.14: XRD pattern of PCC produced at optimum conditions

Approximately (80%) of the world's paper production has been converted to an alkaline paper making process, and precipitated calcium carbonate (PCC) is employed as the production primary filler. An average paper mill may require from about 20,000 to about 100,000 ton per year of PCC (Mathur, 2001, and Tier *et al.*, 2005). In Iraq, the acid process was utilized in paper industry and kaolin is used as fillers. The paper mill now is deadlocked and unfit; therefore, the process must be converted to the alkaline process in the foreseeable future. For that reason the consumption of PCC in paper industry is not considered in this investigation. The state owned companies need about (1500) ton of precipitated calcium carbonate based on the last production year (2002) without considering the production expansions. The non-governmental (private) companies of plastics, paints ... etc consumed about (1500 – 2000) ton/year of PCC, as considered from the importing statistics for calcium carbonate of the non-governmental sector (Jawad, 2007). Therefore, the estimation of local consumption for PCC is about (3500) ton/year. Fig. (15) below shows the precipitated calcium carbonate production process steps sequence.

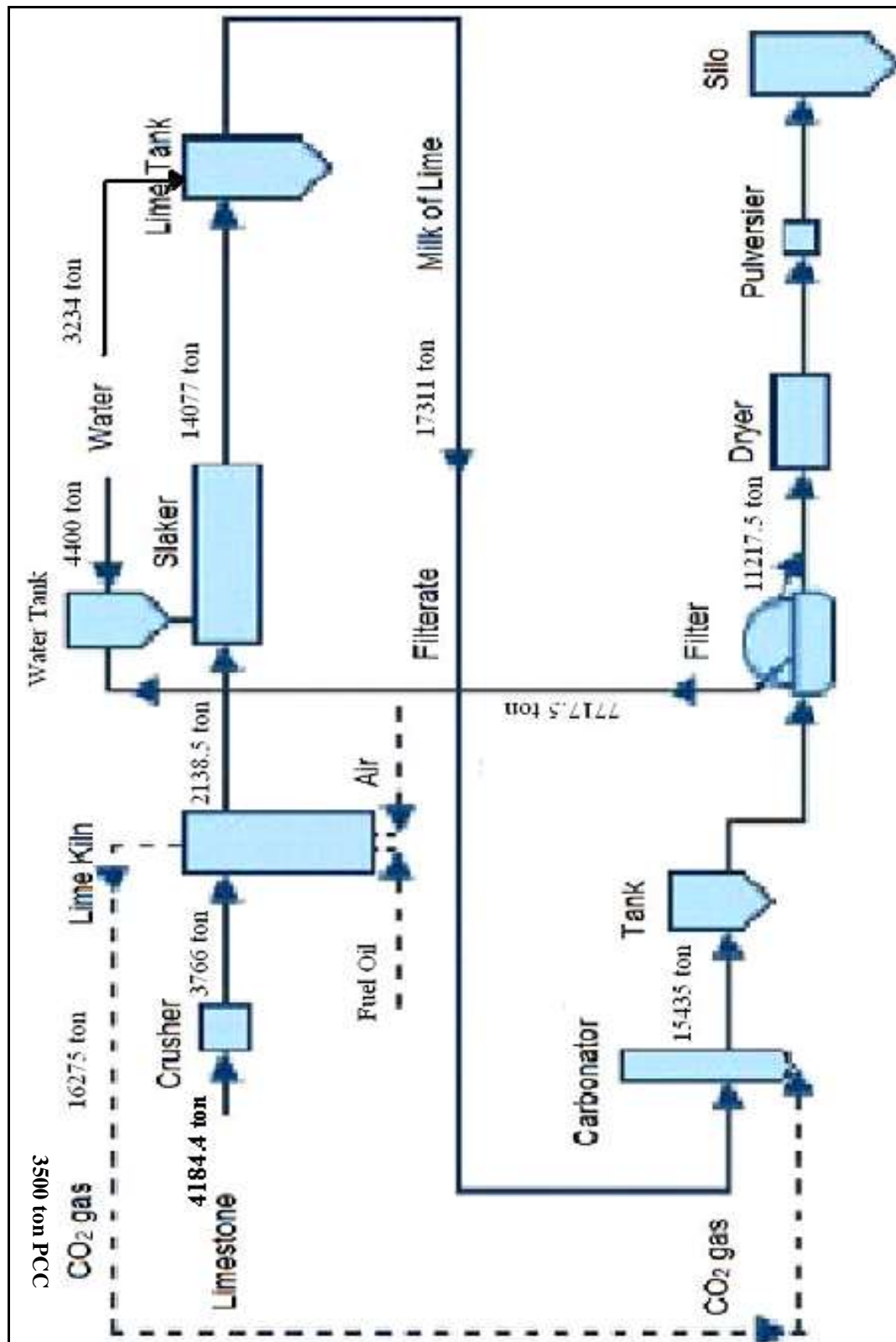


Fig.15: Unit process for precipitated calcium carbonate production

CONCLUSIONS

According to the results of the present bench-scale experiments, the following points can be concluded:

- Complete preparation of quick lime can be obtained by calcining the limestone having particles size of (4 – 10) cm at a temperature of 1000 °C for 1 hour.
- Quick lime is slaked in water at a solid percentage (15%) for 15 – 30 min., and then sieved on 45 micron to remove impurities.
- Precipitated calcium carbonate of high purity white powder (about 99.5%), brightness (95) and particle size about (6 – 12) micron can be produced at the following optimum operating conditions: slaked lime is carbonated with carbon dioxide gas injection at a solid percentage (15%), time (1 hr), CO₂ flow rate (425 lit/hr), and temperature (10 °C).

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