CHAPTER III

EXPERIMENTAL PROCEDURES

Applying phase change materials as a coating on the external surface of the walls of a building as an insulating material was the subject of this research. Using phase change materials in this way is seen as an alternative way to decrease heat and moisture in walls, and consequently reducing the cooling load of the air-conditioners inside the building. Electricity energy would thereby be saved, with the associated cost savings, while maintaining a comfortable internal environment for the inhabitants.

This chapter describes the experimental procedures that were employed in this work. Two major areas of experimentation were undertaken, firstly in regard to the use of waste sugar sediment as a component of Autoclaved Aerated Concrete (creating what we term Improved Autoclaved Aerated Concrete, or Improved AAC), and secondly experiments to test the effectiveness of PCM coating, especially on the Improved AAC, as compared with uncoated building materials; brick, concrete block, and commercial autoclaved aerated concrete. The analysis therefore included both techno-economic and environmental analysis regarding the waste sugar sediment use and effect, and the insulative and thermal transfer effects of using PCM.

The initial experiments using waste sugar sediment as a component of the Improved AAC, tested the replacement of sand and lime in the concrete mix with the waste sugar sediment. Testing various proportions of sugar sediment replacement of the sand and lime resulted in a concrete material that displayed significant improvements in compressive strength, and some improvement in the thermal transfer properties of the new material; Improved AAC. The Improved AAC was selected for the next phase of the experiments, PCM coating, because it is the superior building material.

The investigation regarding the use of PCM as a coating and its effect on the heat transfer properties of the building material consisted of the building of 4 test houses, each with a different wall material i.e brick, concrete block, commercial Autoclaved Aerated Concrete and Improved AAC. A comparison of the economics and the evaluation of life cycle assessment of the Improved AAC was done, and the results are shown in Figure 14.

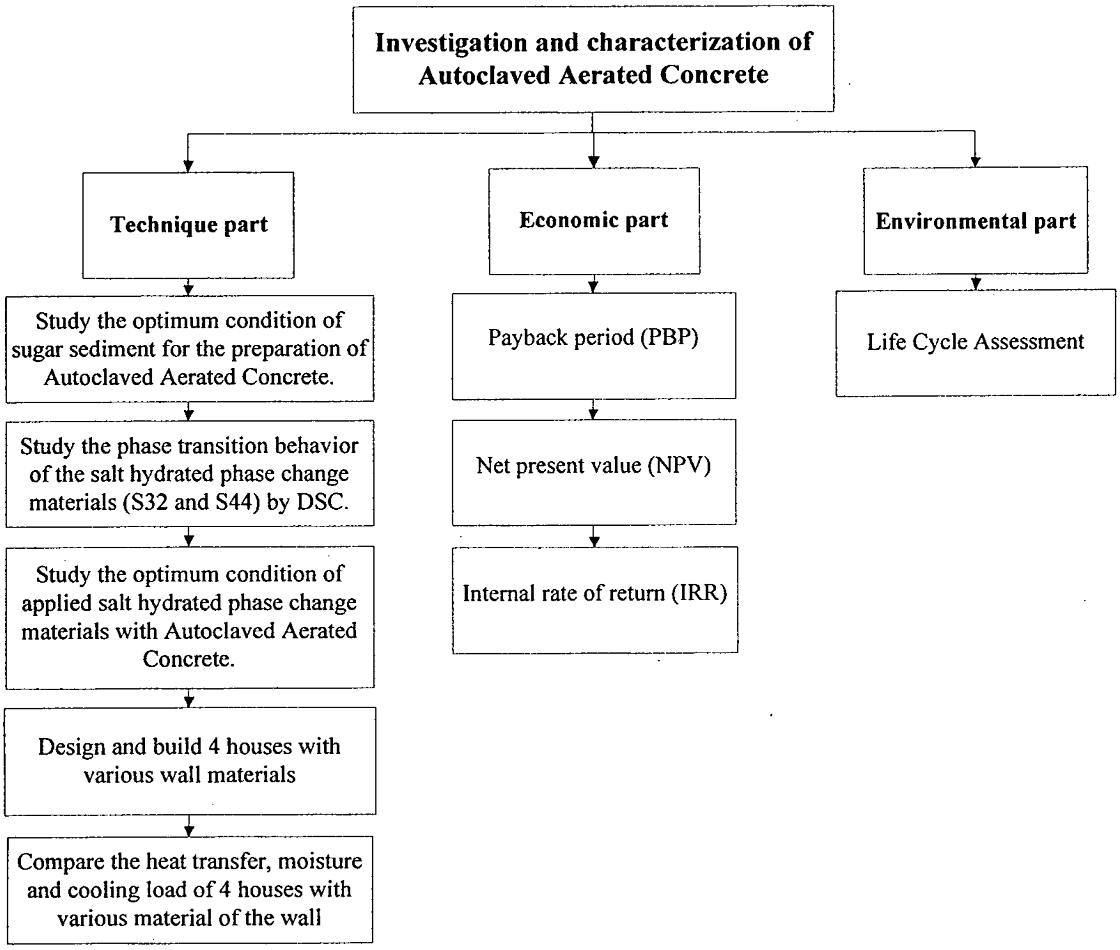


Figure 14 The diagram of experimental procedure.

Materials Used

1. **Development of Improved AAC**
   1.1 Sugar sediment
   1.2 Portland cement
   1.3 Lime (CaO)
   1.4 Anhydrite ($CaSO_4$)
   1.5 Aluminum (Al)
   1.6 Fine sand (less than 90 μm in size)

1.7 Salt hydrated phase change materials

2. Application of PCM

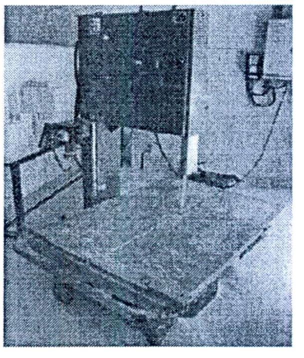
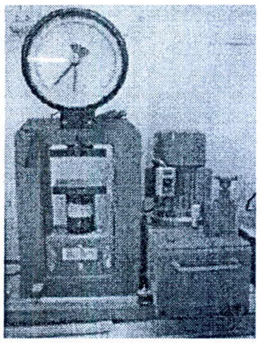
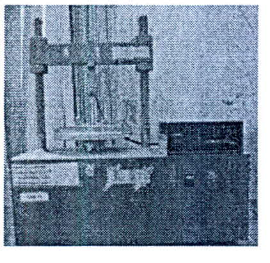
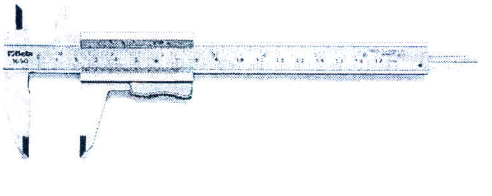
2.1 Brick

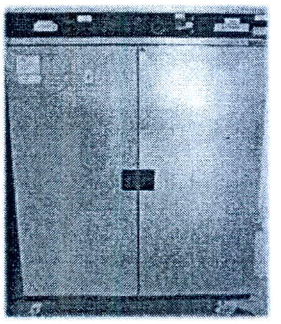
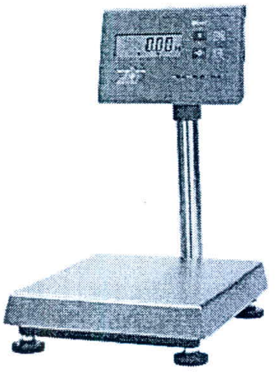
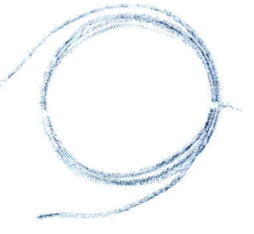
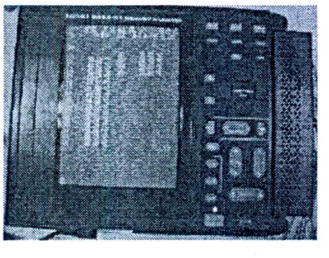
2.2 Concrete Blocks

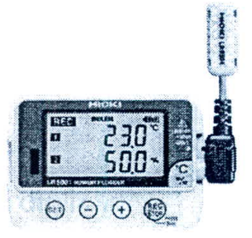
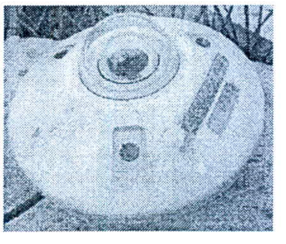
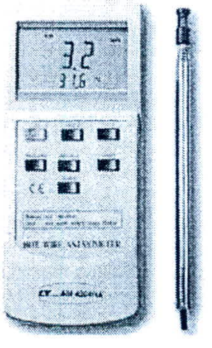
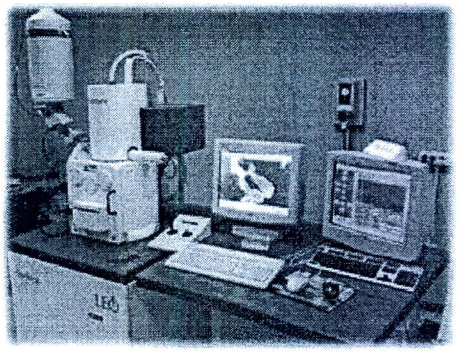
2.3 Commercial AAC

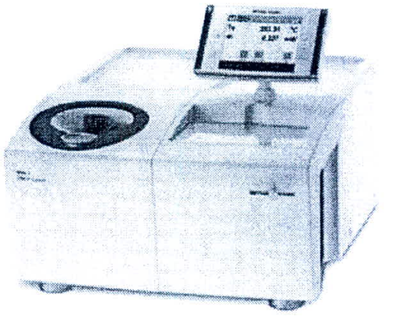
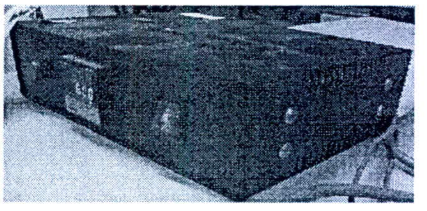
2.4 Improved AAC

Experimental instruments

Lists of the instrument	Images
1. Cutting machine of AAC	
2. Compressive strength machine (Accuracy ±4%)	
3. Flexural strength machine (Accuracy ±0.094%)	
4. Vernier (Accuracy ±0.05mm)	

Lists of the instrument	**Images**
5. Drying oven (Accuracy ±3.1°C)	
6. Digital platform weighting scales (Mettler Toledo) (Accuracy ±0.05 kg)	
7. Digital weighting scales (Accuracy ±0.05 g)	
8. Thermocouple line (Type K); Temperature range -200°C-1250°C (Accuracy ±0.5%));	
9. Data logger (HIOKI8422-51) (Accuracy ±0.8%)	

Lists of the instrument	**Images**
10. Temperature/humidity data recorder (LR5001); Temperature measurement range -40.0 - 85.0 °C; (Accuracy Temperature/±0.5 to ±2.0°C, Humidity/±5%RH to ±10%RH)	
11. Tipping bucket rain collector (Accuracy ±0.2mm)	
12. Pyranometer (Accuracy ±5%)	
13. Hot wire anemometers (Accuracy ±10%)	
14. Scanning Electron Microscopy (SEM)	

Lists of the instrument	**Images**
15. Differential scanning calorimeter (DSC)	
16. Temperature control	
17. Heater	

Experimental procedure in the part of technique

Optimum composition for the preparation of autoclaved aerated concrete

In this work, the AAC preparation will be divided into 3 processes as shown in Figure 15. The first process studied the replacement of fine sand with sugar sediment in various volumes. The resultant product from this process is identified as AAC-S. The second process studied replacement of lime with sugar sediment in various volumes. The resultant product from this process is identified as AAC-L. The third process studied the effects of having replacing different volumes of fine sand and sugar sediment with recycling powder. The resultant product from this process is identified as AAC-SSR.

In commercial AAC production the starting materials are readily available: Lime (CaO), Portland cement, Aluminum (Al), Anhydrite ($CaSO_4$) and fine sand (less than 90 μm in size). Sugar sediment was also available in quantity as a waste by-product of the refining of sugar cane. The usual commercial mixture composition of AAC is Lime (17.17 % by weight), Portland cement (17.876 % by weight), Aluminum

(0.094 % by weight), Anhydrite (2.352 % by weight) and fine sand (62.517 % by weight).

In the first process carried out in this phase of the research, various quantities of fine sand and sugar sediment were first weighed in the required stoichiometric ratio as listed in Table 5. (identified as AAC-S). The content of fine sand was substituted by sugar sediment waste with 0%, 5%, 10%, 15%, 20%, 25%, 30%, 35%, 40%, 45% and 50% by weight with the volume of the other raw materials being fixed.

In the second and subsequent process, various quantities of lime and sugar sediment waste were weighed in the stoichiometric ratio as listed in Table 6 (identified as AAC-L). In this second process, the initial composition was the same as in the first process while the lime content only being substituted by sugar sediment waste with 0%, 5%, 7.5%, 10%, 12.5%, 15%, 17.5%, 20%, 25% and 30% substitution by weight.

Subsequently, in the third process the initial composition was the combined composition of sand, lime and sugar sediment derived from the first and second processes. The lime volume was held constant and sand/sugar sediment volume was substituted with recycling powder at various percentages. The sand/sugar sediment and recycling powder were weighed in the stoichiometric ratio as listed in Table 7. Holding the lime volume constant, the sand/sugar sediment volume was substituted with recycling powder variously at 0%, 5%, 10%, 15%, 20%, 25%, 30%, 35%, 40% and 45% substitution by weight.

In all three compositions, the raw materials were mixed with water in a $0.20 \times 0.20 \times 1$ m mold. All three compositions of autoclaved aerated concrete were fabricated at a temperature of between 180°C -190°C and pressure of 12 bars by a manufacturer certified by Thailand Industrial Standard (TIS) 1505-1998.

Compressive strength, flexural strength, density, water absorption and tobermorite phase measurement

Following the autoclave process, the autoclaved aerated concrete designated as AAC-S, AAC-L and AAC-SSR, from the processes described above, were cut to obtain a uniform block size of $0.075 \times 0.075 \times 0.075$ m. The samples were then dried at 75°C for 24 h and then tested for compressive strength. Other samples were dried at 105°C for 24 h and then examined for density, water absorption and humidity. For testing the flexural strength, all the sample blocks were cut to obtain a uniform size of

0.04×0.04×0.16 m. The samples were then dried at 75°C for 24 h. This process is illustrated in Figure 16. Nine samples were tested in this way. The samples were tested and compared as following Thailand Industrial Standard (TIS) 1505-1998 and Din 4165-1986. The microstructure of the AAC-S, AAC-L and AAC-SSR samples were imaged using the scanning electron microscopy (SEM). X-ray diffraction (XRD) was employed to identify the formation and quality of the tobermorite phase.

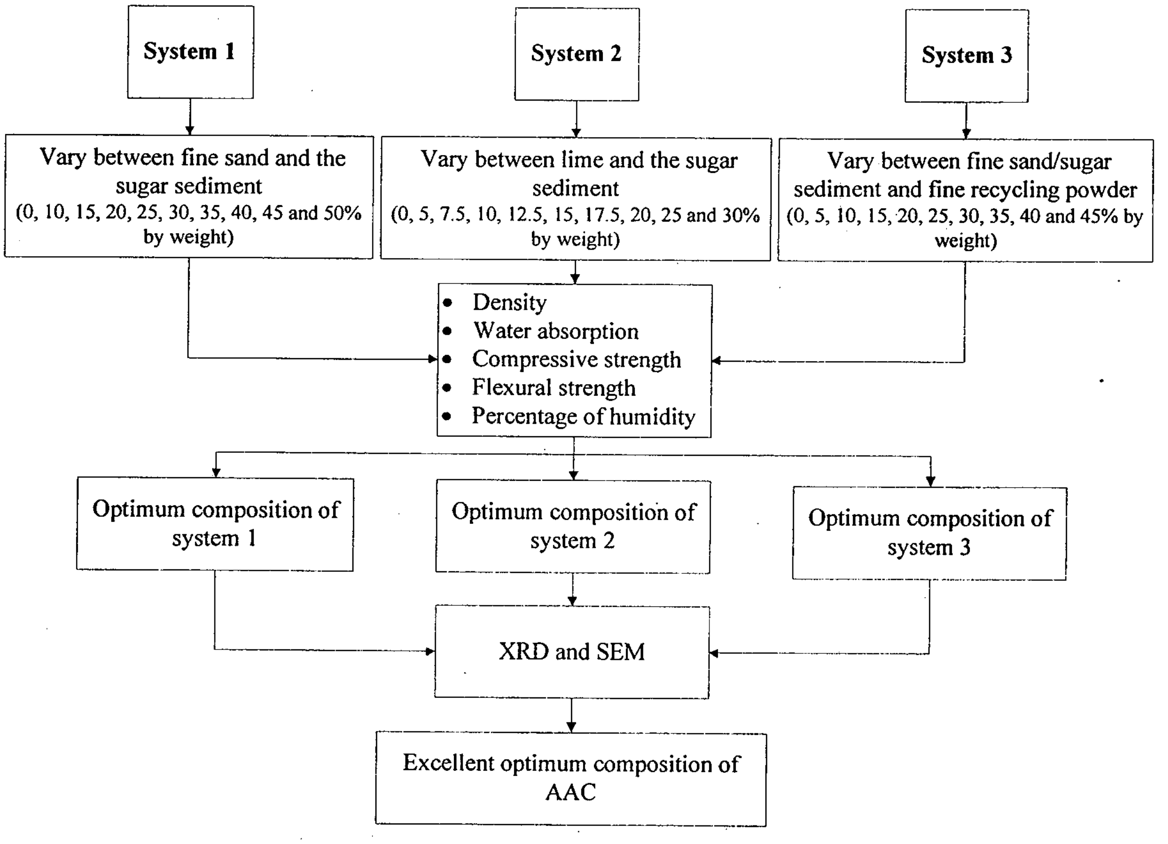


Figure 15 Procedure of optimum composition preparation of AAC.

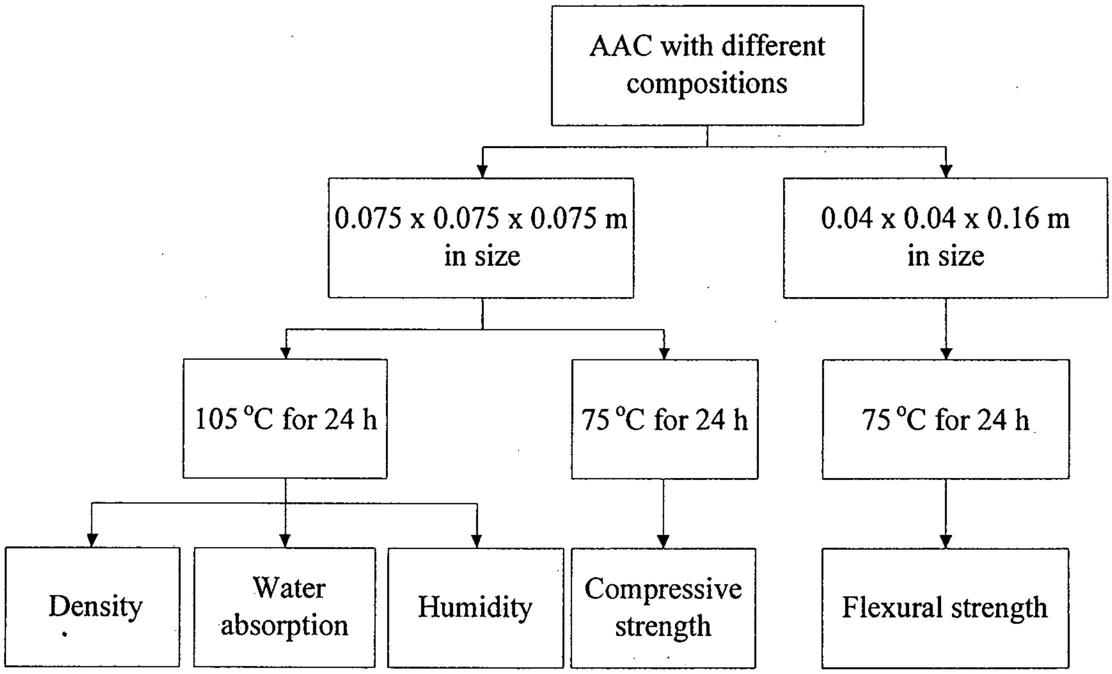


Figure 16 Procedure of physical properties test.

Table 5 Content of raw materials in AAC: the variation between wet fine sand and sugar sediment (AAC-S).

Raw materials	Content of raw materials (weight %)									
	0%	10%	15%	20%	25%	30%	35%	40%	45%	50%
Lime	Held constant at 17.17									
Cement	Held constant at 17.88									
Anhydride	Held constant at 2.35									
Aluminum	Held constant at 0.10									
Fine sand	62.52	56.27	53.16	50.04	46.90	43.78	40.65	37.53	34.39	31.27
Sugar sediment	0.00	6.26	9.36	12.47	15.62	18.73	21.87	24.98	28.13	31.24

Table 6 Content of raw materials in AAC: the variation between lime and sugar sediment (AAC-L).

Raw materials	Content of raw materials (weight %)									
	0%	5%	7.5%	10%	12.5%	15%	17.5%	20%	25%	30%
Lime	17.17	16.32	15.90	15.47	15.04	14.58	14.16	13.73	12.89	12.03
Cement	Held constant at 17.87									
Anhydride	Held constant at 2.35									
Aluminum	Held constant at 0.10									
Fine sand	Held constant at 62.52									
Sugar sediment	0.00	0.85	1.27	1.74	2.16	2.59	3.01	3.43	4.28	5.17

Table 7 Content of raw materials in AAC: the variation among fine sand, sugar sediment and fine recycling powder (AAC-SSR).

Raw materials	Content of raw materials (weight %)									
	0%	5%	10%	15%	20%	25%	30%	35%	40%	45%
Lime	Held constant at 16.11									
Cement	Held constant at 18.11									
Anhydride	Held constant at 2.38									
Aluminum	Held constant at 0.10									
Fine sand	44.35	42.11	39.88	37.68	35.45	33.25	31.02	28.82	26.61	24.39
Sugar sediment	18.97	18.06	17.10	16.16	15.20	14.25	13.29	12.34	11.39	10.44
Recycling	0.00	3.15	6.34	9.48	12.67	15.82	19.01	22.16	25.31	28.50

Phase transition behavior of the salt hydrated phase change materials by the differential scanning calorimeter (DSC).

The second major phase in this project was to test the effectiveness of applying PCM to the external walls of buildings, for insulation purposes. The phase change material in this work was concentrated on the kind of salt hydrate with the type of S32 and S44, which have a melting point of 32°C and 44°C, respectively. This was indexed by the manufacturer. To confirm this result, the endothermic behavior of the salt hydrated phase change materials was investigated by the differential scanning calorimeter (DSC) as illustrated in Figure 17. The result obtained was also compared with the data from the company. The procedure for transition behavior investigation of salt hydrated phase change materials is described:

1. The salt hydrated phase change materials (S32 and S44) about 10-15 mg were finely grinded.

2. Both samples of S32 and S44 were taken into each aluminum pan before analysis by DSC.

3. The samples (S32 and S44) were tested by DSC.

4. The endothermic behavior of the salt hydrated phase change materials was analyzed.

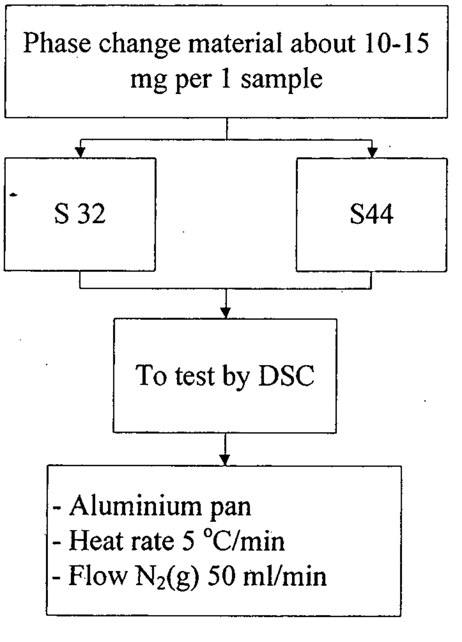


Figure 17 The procedure of DSC analysis

Optimum content of applied salt hydrated phase change materials on the autoclaved aerated concrete surface.

In this process, the heat flux time lag and decrement factor of the original commercial AAC were studied in the 5 samples with different PCM configurations, as defined below.

1. AAC-S0 (Commercial AAC, uncoated)
2. AAC-S0-50 (Commercial AAC with 0.050 kg of PCM-coated external surface)
3. AAC-S0-100 (Commercial AAC with 0.100 kg of PCM-coated external surface)
4. AAC-S0-50C (Commercial AAC with 0.050 kg of PCM-coated external surface and cement-coated surface in both sides)
5. AAC-S0-100C (Commercial AAC with 0.100 kg of PCM-coated external surface and cement-coated surface in both sides)

The procedures are illustrated in Figure 18.

The samples of AAC-S0, AAC-S0-50, AAC-S0-100, AAC-S0-50C and AAC-S0-100C were set up in the processes illustrated in Figure 19 and Figure 20. To test the temperature at different positions and thickness of samples, the thermal source (heater) was controlled at the temperatures of 40°C, 50°C and 60°C. This temperature range was considered as the wall temperatures of most buildings in Thailand are in the range of 40-60 °C [81, 82, 83].

The same process was then applied to samples of Improved AAC. That is

1. AAC-SSR (Improved AAC, uncoated)
2. AAC-SSR -50 (Improved AAC with 0.050 kg of PCM-coated external surface)
3. AAC-SSR -100 (Improved AAC with 0.100 kg of PCM-coated external surface)
4. AAC-SSR -50C (Improved AAC with 0.050 kg of PCM-coated external surface and cement-coated surface in both sides)
5. AAC-SSR-100C (Improved AAC with 0.100 kg of PCM-coated external surface and cement-coated surface in both sides)

The procedures are illustrated in Figure 18.

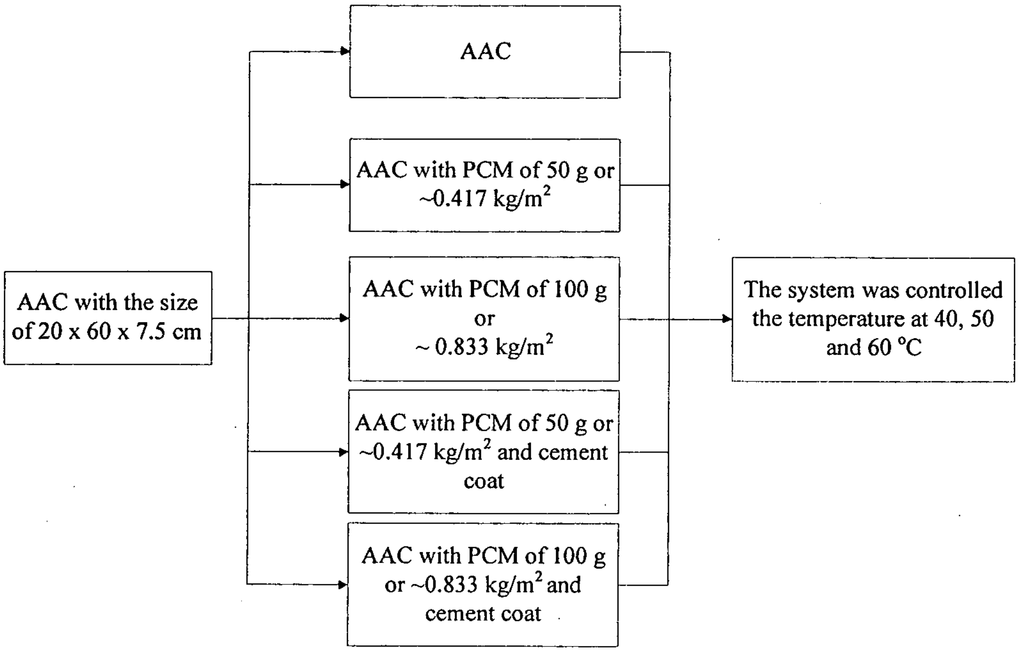


Figure 18 Procedure of optimum PCM content study for AAC.

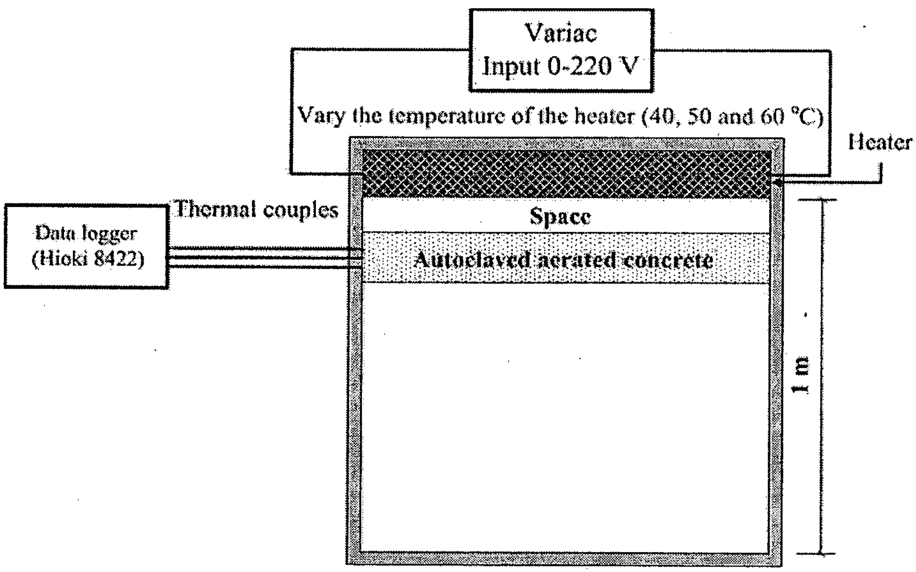


Figure 19 Schematic diagram of the experimental set up.

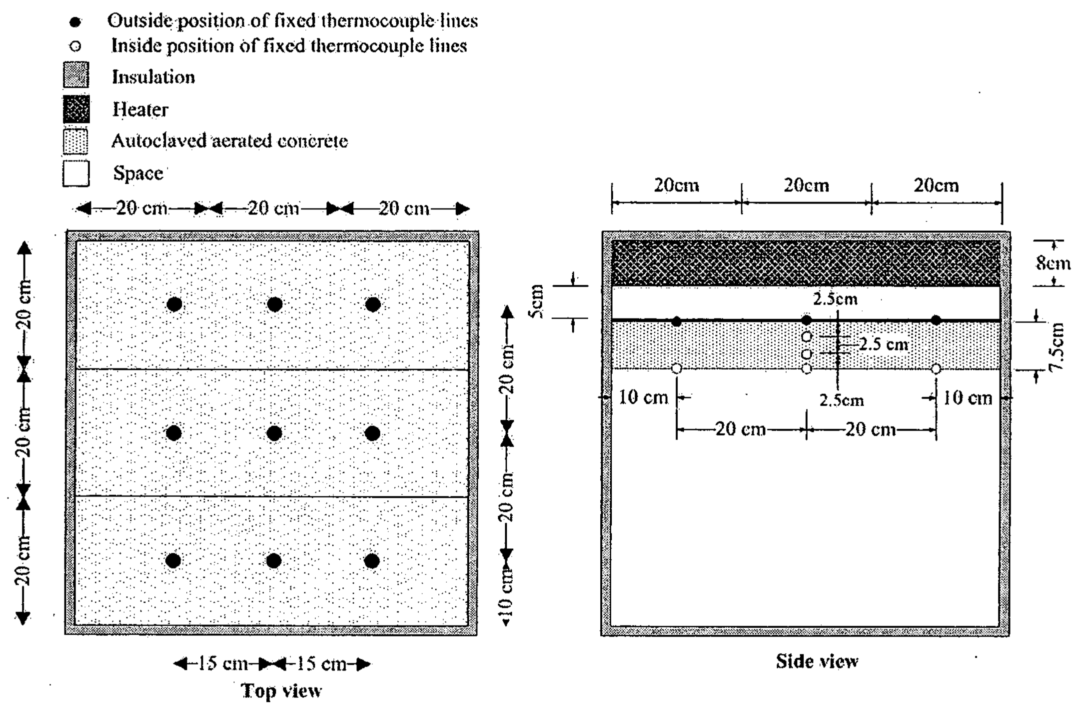


Figure 20 The thermal testing position of AAC.

Design and build the 4 test houses with various wall materials.

The houses were designed and built for testing the use and thermal properties of various wall materials in the tropical climate. The elevation diagram of the houses is shown in Figure 21. This also included the use of PCMs on the Improved AAC construction material.

The walls of the four houses were constructed from the brick, concrete block, commercial autoclaved aerated concrete and the improved autoclaved aerated concrete. Each house was 13.25 m^3 in volume, with 4 walls, each wall 5.52 m^2 in area and 0.08 m thick with a coating of cement plaster. All wall interior and exterior surfaces were unpainted. The roofs of the four houses used CPAC Monier with aluminum foil to reflect heat. The inclination of the roof angles was 30° to the horizontal plane. The ceiling of each house was made of gypsum board with thickness of 0.01 m to prevent or reduce heat being transmitted through the roof. Each house had a wooden door 0.84×1.84 m in size which was set in the south side of each house. A glass window 0.72 m wide ×1.0 m high was set in the west side of each house. The dimensions of each house were:

1. Each house was a square 2.40 m x 2.40 m.
2. Wall height 2.30 m.
3. Door 1.84 m high by 0.84 m wide.
4. Roof height above the eaves 1.00 m.
5. The width of eaves 0.40 m.

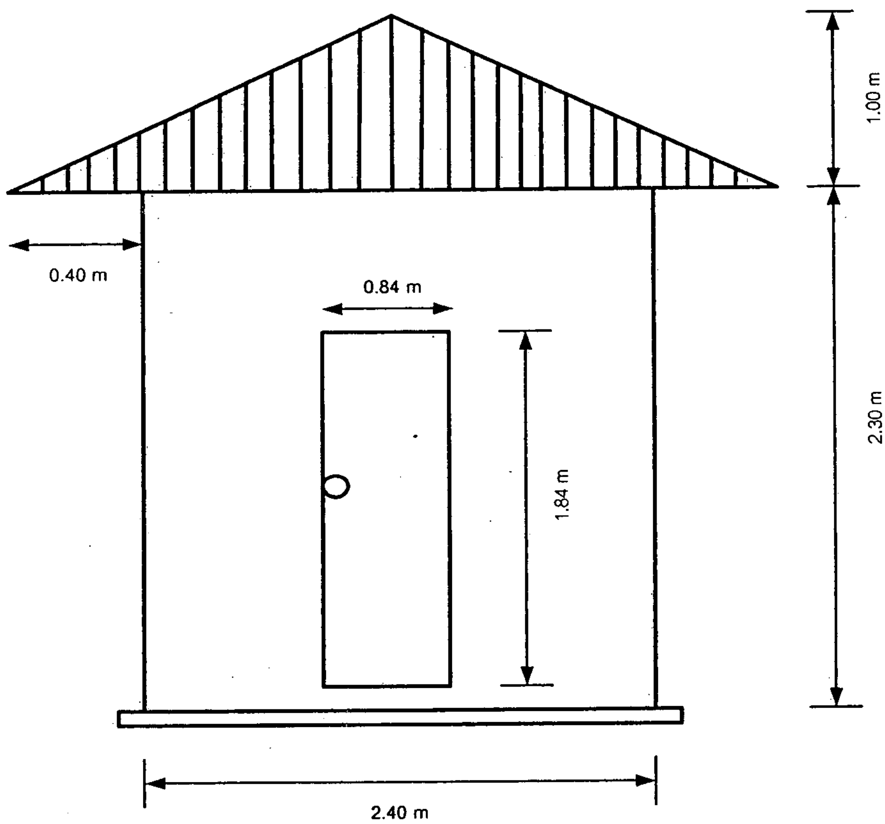


Figure 21 House pattern for testing

Test the actual use of 4 houses with various wall materials.

The four houses were tested in daytime conditions and at night to study the endothermic and exothermic behavior of the walls over the period of 24 h. The test procedure is described below:

1. A pyranometer, anemometer and rain gauge were set in the outdoor area adjacent to the houses to measure the intensity of solar radiation, wind speed and rainfall.

2. Temperature/humidity data recorders were set both inside and outside each house to measure the temperature and humidity of both the interior of each houses and the exterior ambience.

3. K-type thermocouples with accuracy of ±0.5% were employed to measure temperatures. They were attached to both the exterior and interior wall surfaces on all 4 sides by thermocouples in close contact with the wall surface and insulated using aluminum foil tape. Thermal paste was applied to ensure good thermal contact between the thermocouples and the surfaces as illustrated in Figure 21. Temperature data of all inside and outside walls, the ceilings and the roofs of each house were logged using the data logger.

4. The intensity of solar radiation, humidity and temperature in all positions were measured and recorded from 6am of day to 6am of the following day, giving the 24 h test cycle.

5. All data were recorded at 5 min intervals using a data logger.

6. For this investigation was undertaken in the summer season and in a simulated rainy season.

7. In a simulated rainy season, the simulated rainfall level in this study was considered from mean annual rainfall in Thailand in last 10 year period (2004-2013).

8. The cooling load of the air conditioners was tested continuously over each 24 h period with closed windows and doors in each house. The interior of each house was maintained constantly at around 25 °C for the purpose of load testing the air conditioners.

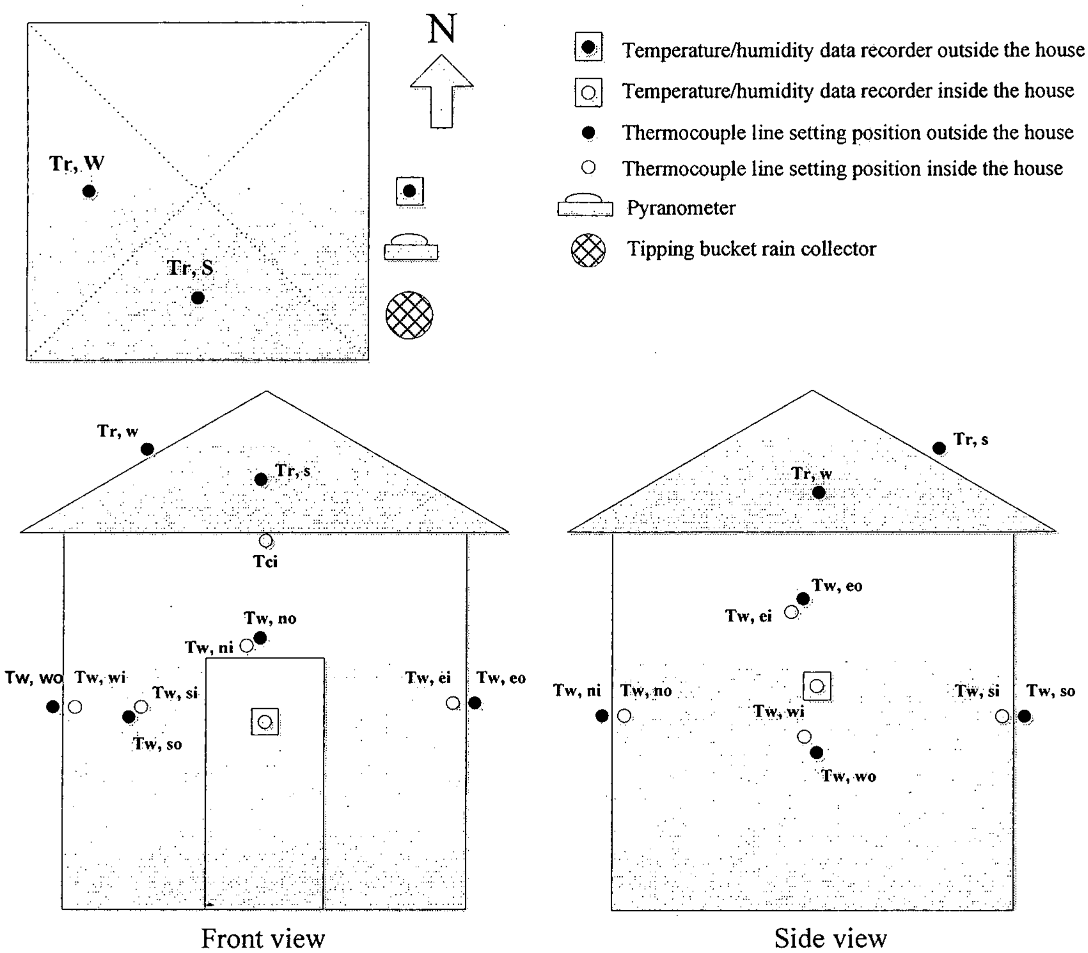


Figure 22 The setting position of the thermocouple lines of 4 houses.

Table 8 The position of the thermocouple lines for the actual use test

Symbol	Meaning
Tw,ni	The north inside wall temperature
Tw,wi	The west inside wall temperature
Tw,si	The south inside wall temperature
Tw,ei	The east inside wall temperature
Tw,no	The north outside wall temperature
Tw,wo	The west outside wall temperature
Tw,so	The south outside wall temperature

Table 8 (cont.)

Symbol	Meaning
Tw,eo	The east outside wall temperature
Tr,w	The west roof temperature
Tr,s	The south roof temperature
Tci	Ceiling temperature
Tai	Room temperature
Tao	Ambient temperature

Experimental procedure in the part of economics

The standard methods of economic analysis of an investment made in any course of action include calculation of Net Present Value (NPV), the Payback Period (PBP) and Internal Rate of Return (IRR) [84]. An economic analysis comparing the commercial concrete and the improved autoclaved aerated concrete using these parameters was undertaken. The calculation in regard to these parameters was only studied in the summer.

Payback period (PbP)

Payback period is a measurement of the period of time necessary for the investment to pay for itself. That is, the sum of the periodic net profit values over time. PBP is given in the following equation:

$$PBP = \frac{A_o}{A_s} \quad (3.1)$$

Net Present Value (NPV)

Net Present Value is the difference between the present value of cash inflows, or savings in cash outflows and the present value of cash outflows. The initial investment is considered to be the net outflow where the project does not incur further cash outflows. The Present Value of the cash inflows or savings in outflows over the forecast period of time is calculated by applying a discounting factor to take into consideration the future remoteness of the potential inflows or savings. For an investment to be considered worthwhile, the discounted inward cash flow must exceed the discounted outward cash flow. The initial investment amount is not discounted, as it occurs at $Time_0$. An indifferent analysis implies inflows equaling outflows on a discounted basis. This indifference between acceptance and rejection of a project is excluded from definition as following equation below:

$$NPV = -A_o + A_s\frac{[(1+i)^n-1]}{i(1+i)^n} \tag{3.2}$$

Internal rate of return (IRR)

The Internal Rate of Return is defined as the discount rate that causes the net present value of a cash flow to equal zero. It is calculated as follows:

$$IRR = 0 = NPV = -A_o + A_s\frac{[(1+i)^n-1]}{i(1+i)^n} \tag{3.3}$$

Where A_o = Increasing of the investment when compared with the brick (bath)

A_s = Electrical cost saving (bath)

i = Discount rate (%); (Loan Rates of Bank of Ayudhya as of 25 April 2014 = 7.75%)

n = Life cycle (yr); (n = 25 years)

Experimental procedure in the part of environment

Goal and scope definition

To study and assess the environmental impacts associated with all the stages of the autoclaved aerated concrete's life (G4 type with 0.20x0.60x0.075 m in size). This was analyzed by SimaPro version 7.2 from the raw material extraction through materials processing, manufacture, distribution, use, repair and maintenance, and disposal or recycling as shown in Figure 23.

Functional Unit

The functional unit of the autoclaved aerated concrete was the production of autoclaved aerated concrete which was defined by 1 kg/m^3.

System boundaries

Method for the life cycle assessment of the autoclaved aerated concrete (G4 type with 0.20x0.60x0.075 m in size) was considered from cradle to grave (i.e. from the resource extraction to materials processing, manufacture, distribution, use, repair and maintenance, and disposal or recycling).

Life cycle inventory

Life cycle inventory (LCI) involved the compilation and quantification of the material and resource inputs and outputs for a given product system throughout the life cycle of a product, in this case autoclaved aerated concrete. LCI is a "cradle to grave" calculation of the environmentally significant inputs and outputs of a system. The environmental burdens measured in this case study include raw material input requirements, total energy and consumed fuel, released air and water emissions, and total solid wastes associated with the product's life-cycle.

Cut off

Some data for the life cycle inventory analysis of the autoclaved aerated concrete process was ignored if its content is less than 5 % when compared with the required product. These data will be not considered in this study.

Reference

The life cycle inventory of the autoclaved aerated concrete was collected from the primary data of INSEE Superblock Company Limited between January, 2012 and December, 2012.

Procedure of life cycle assessment

1. Collect the data about the energy use, raw materials, and waste from the manufacturing process, as shown in Figure 23 and Figure 24.

2. Take the received data to manage the life cycle inventory as following the ISO 14044:2006.

3. Anayze the data from the life cycle inventory for its environmental impact using SimaPro version 7.2.

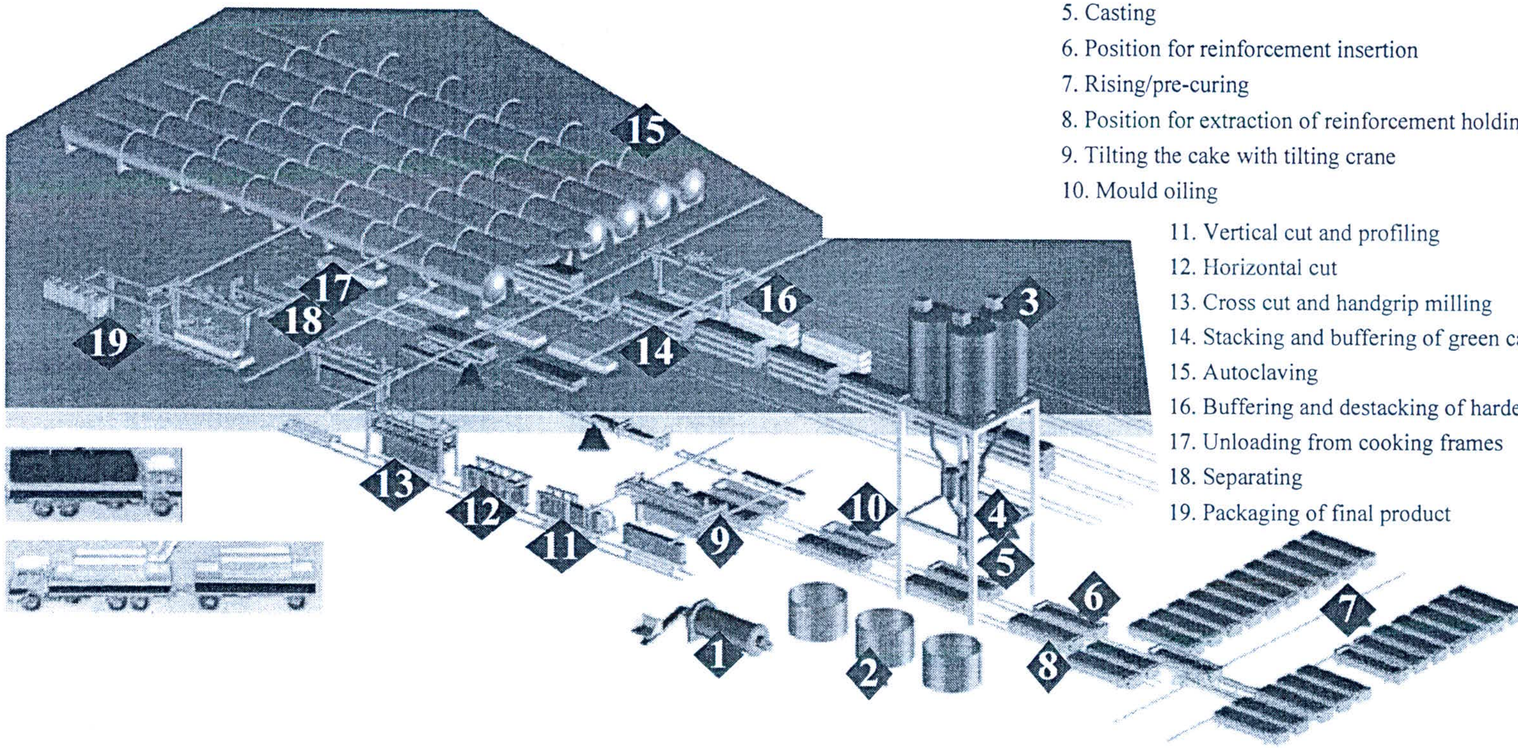
4. Analyze the environmental impact assessment in the process of buildings or homes construction, residential applications and repair using SimaPro version 7.2 and compare the results between the commercial autoclaved aerated concrete wall and the improved autoclaved aerated concrete wall.

Procedure of autoclaved aerated concrete manufacturing

To evaluate the life cycle of autoclaved aerated concrete, the manufacturing process as shown in Figure 23 was investigated and explained following below:

The raw material preparation process of the concrete includes finely wet-grinding of a quartz sand and water mixture, in a ball mill (1). The fine sand slurry is then brought into the slurry tanks (2). Lime, cement, gypsum and aluminum powder/paste are kept in silos (3). They are then mixed together in pre-determined proportions (4). The mixed raw materials are poured into the moulds (5). The moulds with the mixed raw materials are moved to a position for the insertion of reinforcement holding pins (6) and then to pre-cure the cake for about 120-180 minutes in the rising area (7) (in the pre-curing process the cake in the mould rises due to the reactions of the raw materials creating gas bubbles within the mixture) which is then ready for subsequent cutting. The moulds are then moved to the position for extraction of the reinforcement holding pins (8) and then to the tilting crane (9). The moulds are tilted by 90° the tilting crane, which unlocks each mould and removes the mould body, so that the cake remains on the cutting platform. The cake is then cut into the required sizes according to market requirements or customer orders. The cake is cut by high precision cutting machines, using pneumatically tensioned cutting wires. The pre-cutter and vertical cutter cut the block length and panel width. Profiling (tongue and groove) can be cut into the cake with profiling knives (11). The horizontal cutter cuts the block and panel thickness (12). The cross cutter cuts the block height and the panel length (13). The cakes are then stacked on cooking frames (14) and are then fed into the autoclave, where they are cured for 10-12 hours at a temperature of 180-190°C with saturated steam at a pressure of 12 bars (15). After the autoclaving is completed, the cakes are unloaded from the cooking frames and restacked (15). Depending on the product specification and the raw material quality, it may be necessary to separate the layers (18). The finished products are packed, usually on wooden pallets, according to market requirements (19).

The moulds are prepared for reuse by being cleaned and coated by oil (10). The mould restarts the cycle in no.1-10.



1. Ball mill for milling sand
2. Slurry tanks
3. Silos for lime, cement and gypsum
4. Dosing and mixing
5. Casting
6. Position for reinforcement insertion
7. Rising/pre-curing
8. Position for extraction of reinforcement holding pins
9. Tilting the cake with tilting crane
10. Mould oiling
11. Vertical cut and profiling
12. Horizontal cut
13. Cross cut and handgrip milling
14. Stacking and buffering of green cakes
15. Autoclaving
16. Buffering and destacking of hardened cakes
17. Unloading from cooking frames
18. Separating
19. Packaging of final product

Figure 23 Autoclaved aerated concrete production procedure

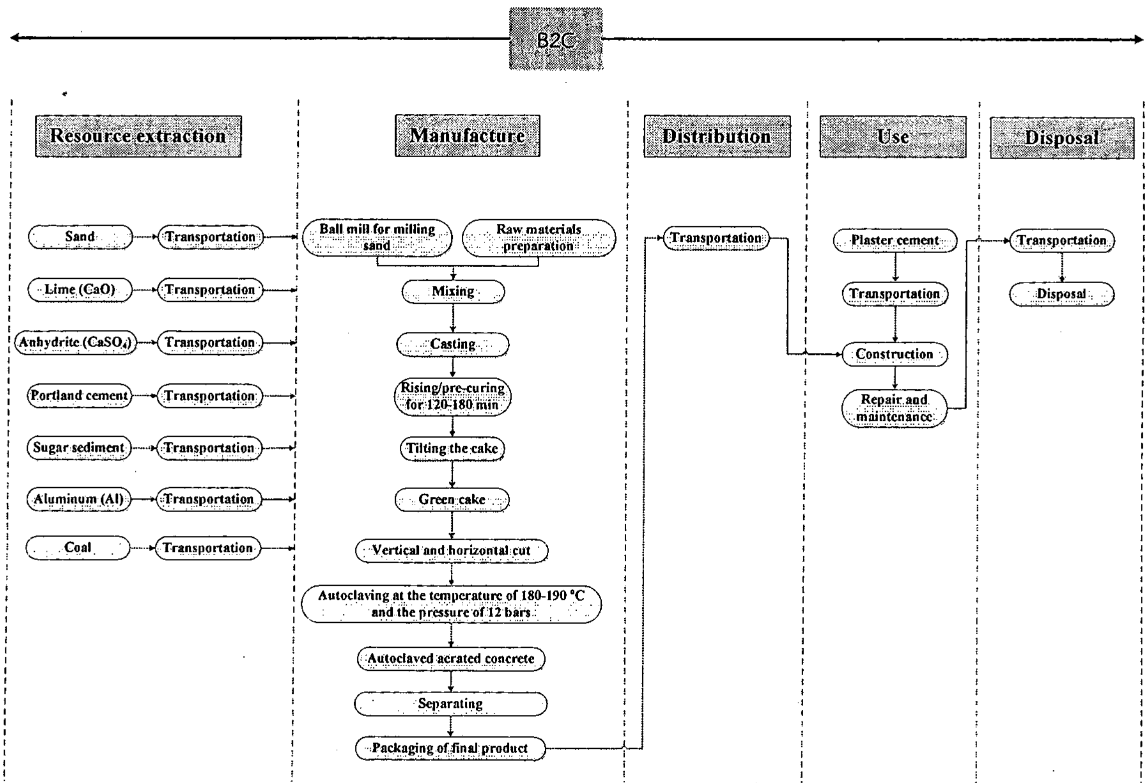


Figure 24 Life cycle process flow diagram: Autoclaved aerated concrete (G4 type with 20x60x7.5 cm in size).