Impregnation of calcium chloride on activated carbon surface

This performance of solar refrigerator is based on some key parameters like ammonia carrying capacity, adsorption rate, heat transfer mechanism, stability etc., this paper presents the stepwise detailed preparation of consolidated calcium chloride impregnated on the surface on activated carbon. Ammonia was charged into the tube containing blocks under high pressure of 7 bars was applied during initial charging session and total adsorption was noticed in 25 minutes and the process was repeated. The total amount of ammonia adsorbed was 2. 4 kg and was capable of taking more ammonia. Agglomeration effect was totally avoided by the even distribution of calcium chloride on the surface of activated carbon. Carbon was activated at elevated temperature of 700 C for optimum properties.

1. Introduction

Solar refrigeration and heat pump are machines that work on wide range of temperature, especially when ammoniates or ammonia derivatives are involved as sorbents then temperature can range from -50oC to 300oC [1]. Furthermore it is environmentally benign and it does not contribute to global warming or ozone layer depletion. They do not usually utilize CFCs or HCFCs as refrigerants.

This type of refrigeration is based on the phenomena of adsorption which is a surface phenomenon in which one is adsorbent and second is adsorbate or refrigerant. Two main types of adsorbents are used, one is physical adsorbent like activated carbon in which the adsorbate/refrigerant is adsorbed on its surface by week Vander Waals forces. This process is dependent on the surface area of adsorbent. It is said that one gram of

activated carbon has an area equal to the area of a tennis yard. The mass of adsorbate/refrigerant adsorbed in this process is very less because of weak Vander Waal forces. The second type of adsorbent is chemical adsorbent like Calcium chloride. When it comes to sorption capacity we use chemical adsorbents. The mass of refrigerant incorporated here could be as high as 1. 05 kg/kg salt [2]. But there are two serious concerns with this, one is granular salts have very low thermal conductivity and second is the agglomeration phenomenon. The salts after some cycles of adsorption and desorption it undergoes it swells, compacts and agglomerates, which leads to drastic reduction of the surface area and ultimately reduces the adsorption capacity. Wang et al. [3] mixed activated carbon with calcium chloride salt which overcame the phenomenon of agglomeration and observed a constant adsorption capacity. Lu et al. [4] observed speciï¬c cooling power ranging from 111. 2 to 865. 8 W/kg using adsorption ice maker. Vasiliev et al. [5] used carbon fiber with calcium chloride and reported to have increased heat and mass transfer phenomena and overcome agglomeration. Later Mauran et al. patented a process to make impregnated consolidate blocks of calcium chloride and activated carbon. Han and Lee [6] found the thermal conductivity of different salts impregnated in expanded graphite blocks and measured values close to 50 W m-1 K-1 when the amount of expanded graphite was 70%.

Ammonia is used as refrigerant and the process of chemisorption is governed the following two basic equations.

1. $CaCl_2 . 2NH_3 + 2NH_3 \rightarrow CaCl_2 . 4NH_3$

2. $CaCl_2 . 4NH_3 + 4NH_3 \rightarrow CaCl_2 . 8NH_3$

This process of impregnation of calcium chloride on the surface of activated carbon not only ensures uniform distribution of calcium chloride on the surface of activated carbon but also avoids agglomeration over many synthesis cycles. Balat and Spinner [7] reported that the texture of expanded graphite favors the mass transfer of the adsorbate/refrigerant which leads to improved kinetics when activated carbon combined with a salt is used in the chemisorption system.

Experimental Setup and Results:

The following steps were performed during formation of composites blocks of calcium chloride and activated carbon:

Heat treatment of calcium chloride and activated carbon, Making solution of water and calcium chloride and mixing it with expanded graphite, Impregnation of calcium chloride in expanded graphite Calcination of hydrated calcium chloride into calcium chloride, Grinding of the dried impregnated mixture of calcium chloride and activated carbon and finally formation of consolidated blocks from powder obtained from grinding.

Heat treatment of carbon:

In the experiment performed by Wang et al [6] the expanded graphite is heated at the temperature of 300oC. But according to Han et al the temperature of the heat treatment influences the degree of expansion and these authors stressed on the importance of expanded graphite at temperature higher than 600oC to ensure a proper expansion. Han and his

co-worker [9] also noted that the graphite expanded at temperatures above 700oC had the lowest densities and at least twice the porosity of expanded graphite treated at 500oC. The expanded graphite used has the chemical composition given in table.

The heat treatment of carbon powders was carried out to increase its porosity and to remove the contaminants from the carbon powders. Carbon powders were placed in container and placed in an electric oven at 100oC for 7 hours in order to remove the moisture contents contained in it. 2kg of carbon powders were placed in two separate aluminum vessels in the oven at 100oC for 7 hours. After this it is placed in desiccators to avoid absorption of moisture contents till the start of the second process. In the last step heat the carbon powders obtained from the first step in vacuum at 700oC in a tube furnace for a dwell time of 14 minutes.

Procedure:

Put small amount of carbon powders in blind stainless steel tube of diameter 1. 5 inches and length 15 inches and press them with a rammer so that the tube can hold a maximum amount of carbon powders. After placing the carbon powders in the tube seal the open end of the pipe with wire gauze with …… mesh and cotton cloth in order to prevent sucking of carbon powder by vacuum pump connected through a plate at the open end, tight the end cap of the tube to avoid infiltration of air into the tube. If air enters the tube in the heating process underway simply oxidation of the powder takes place. Then Place the tube in the tube furnace and connect it with the vacuum pump to remove the air entrapped in the tube and any other

residual gases produce during heating of carbon powders. In the experiment by Wang et al. [2] expansion of graphite above 700 C has no significant effect on the reduction of the density of activated carbon also Hans [3] and coworkers shows that carbon expanded at a temperature of 700 C has lowest density and twice the porosity as compared to expansion at 500 C, so the temperature of the furnace is set at 700 oC giving a ramp rate of 10 oC/minute. When the temperature of the furnace reached 700 oC we set the machine at a dwell time of 14 minutes in order for carbon powder to undergo full expansion. After that the furnace was turned off and the tube was allowed to cool down. When the furnace cools down remove the carbon powders from the tube and stored it in a desiccator. The furnace takes one and a half hour to reach 700oC and 2 hours for cooling the tube as a result this process took us a lot of time. About 150 grams of carbon powder were expanded in a single run. This process was repeated until a total mass of 2. 4 kilograms of expanded graphite was obtained.

Tube used for holding the carbon powder in tube furnace:

A tube was designed to hold the carbon powders in the tube furnace. The tube was made of stainless steel which can withstand high temperature of the tube furnace. A metal plate is welded to one end of the pipe to permanently close one side of the pipe and the other end was left open for pouring the powders into the tube. A cap was made to seal the open end of the pipe after filling with the carbon powders. The cap has an extension pipe for connecting the tube with the vacuum pump. A 300 grid mesh was used before the cap and cotton cloth was also applied to avoid the suction of carbon powders into the vacuum pump. The cap itself is made of two metal

plates. One plate has a groove of 3 mm depth and a diameter equal to the pipe diameter which holds the tube in place. The two plates were bolted with each other through four nuts and bolts.

The specifications of the tube are given below;

Total length30 in

Diameter of tube1. 5 in

Volume of tube0. 000376m3

Density of carbon450 kg/m3

Mass of carbon0. 1693 kg

Table. 2 dimensions of vacuum tube

Heating of calcium chloride:

Heat treatment of calcium chlorides was also carried out remove the moisture content and any contaminants in it.

2. Experimental procedure:

The following steps are carried out during the heat treatment of the calcium chloride.

Since calcium chloride salt is very hygroscopic in nature and it absorb moisture very easily. Put 4kg of calcium chloride salt in stainless steel vessels and covered the mouths of the vessels with aluminum foils. Small

holes were made in aluminum foil for the moistures and gases to escape out of the vessels. These vessels were then placed in an oven at a temperature of 100oC for 10 hours to remove the moisture contents out of the calcium chloride salt. After heating calcium chloride salt at 100oC. It is then placed in desiccators to avoid the absorption of moisture by the salt until next step.

Preparation of solution of calcium chloride and activated carbon using distilled water:

After the heat treatment of calcium chloride and activated carbon 60% salt solution of calcium chloride in distilled water is prepared. 4kg of calcium chloride and about 7 liters of distilled water was used for making solution. Activated carbon is then stirred thoroughly in bottle and poured into the solution of calcium chloride salt and stirred for some time for uniform mixture. Based on the result of Wang et al. [6] best performance is shown when calcium chloride and expanded graphite are mixed in the ratio of 4: 1.

Figure: 60% solution of calcium chloride and water

Figure: mixture of solution of calcium chloride and activated carbon

Impregnation of calcium chloride in expanded graphite:

The solution is then dried for 10 hour at 110 0C in an electric oven to remove free water and allow

Impregnation of calcium chloride in expanded graphite.

This figure shows the final shape of uniform mixture of activated carbon and calcium chloride salt when it loses all water in an electric oven.

Calcination of hydrated calcium chloride into anhydrous calcium chloride:

After impregnation of calcium chloride on the surface of expanded graphite the solution is again heated in an oven at 250oC for 7 hours [] to calcinate the calcium chloride as a result the hydrated calcium chloride become anhydrous.

$CaCl_2 . nH_2O \quad CaCl_2 + nH_2O$

Grinding

When the solution is dried and dehydrated in the electric oven the mixture is grinded in the grinding machine to make fine powder of the composite materials. The fine powders are then placed in desiccators in order to avoid the absorption of moistures by the powders until the next process.

Die used for making consolidated blocks:

In order to compress the obtained the powder above under pressure of 10 MPa using UTM it is placed in the mold shown below. The die consists of a cylindrical tube, a piston, a metal plate at the bottom of cylindrical tube and stainless steel rod. The cylindrical tube, metal plate and piston is made of mild steel. Graining of piston and cylindrical tube is done to make the surface smother so that block would not stuck in it after compression under immense pressure. No binder was used in the compaction of powder into consolidate blocks.

- Specification of cylinder are given below
- Thickness of cylindrical tube is 5mm and its height is 152. 4 mm.
- Internal diameter is 50 mm.
- Specification of piston are given below
- Height is 15 in
- Diameter of piston is 49mm
- The piston having a hole at its center is lengthwise whose inner diameter is 15 mm.

Figure: Die for making consolidated blocks of calcium chloride and activated carbon

Consolidate blocks Formation:

To make hollow cylinder of obtained powder under UTM following steps were followed.

The resulted powder (impregnated with calcium chloride) are poured into the cylindrical part of the die through funnel, then a piston is placed on the top and a small load is applied manually to make the powder a bit compressed. The die is fitted in a UTM and pressed under 10MPa of load. [10]

Figure: composite blocks of calcium chloride and activated carbon

The composite blocks are placed inside the test unit, experiments were performed and test results were analyzed.

RESULTS AND CONCLUSIONS:

EPERIMENTAL PROCEDURE FOR CHARGING

Charging of ammonia required great care because it is very dangerous and can cause blindness and even death when come in contact with human. Therefore during charging great care should be taken and all the safety rules must be followed.

Before charging ammonia make sure that entire setup is leak proof and there is no leakage during charging or after charging. Compressor is connected to the generator line and whole system is evacuated from air and it’s noted that either vacuum is created or not by taking reading through pressure gauges attached to generator line. By performing these entire tests and conforming that no leakage is present the system is passed through the following steps.

s. no. Initial pressure before chargingFinal

pressure after chargingTime taken until 0 barMass adsorbed

1st run-1 bar6. 0 bar25 min600 grams

2nd 0 bar7. 0 bar39 min700 grams

3rd 0. 4 bar8. 0 bar30 min500 grams

4th 0. 5 bar10 bar49 min750 grams

1. First of all generator line is heated for 3 hours at 100oC with the vacuum pump connected to remove the gases adsorbed in the adsorbent. This process is called degasing.

2. Secondly the system is evacuated using vacuum pump and it is placed idle for 30 minutes to find out whether any leakage is occurring or not.

3. After the system is evacuated the compressor along with all plumbing is detached from generator line and ammonia charging hose is placed in place and tight fit is ensured.

4. Then charging line is connected with ammonia cylinder. The valve pipe connecting the ammonia tank and generator line is supplied with a manual valve. Initially the valve is opened slowly to flood the ammonia into the generator line, the pressure inside the generator line starts increasing. As ammonia starts adsorbing inside the pipe the pressured reduces while the manual valve is opened and closed at the succession of the processes.

5. After charging the system is closed using manual value to stop the further flow of ammonia and wait until the pressure in the system reduces due to adsorption of ammonia in the adsorbent material.

6. When the pressure reaches its minimum level the system is charged with ammonia again and wait until the pressure in the generator line decreases.

7. This process is repeated until 2. 4kg of ammonia is charged into the system.

The experimental data achieved during charging of the system is given in table given

The total amount of ammonia charged into the system was about 2. 4 kg.

The mass of ammonia was find out using digital scale. The generator line is placed on the scale and mass is noted before and after charging. In this way mass of ammonia adsorbed is find out.

CONCLUSIONS:

- The powders were successfully activated using available research facility.
- Impregnation of calcium chloride on the surface of activated carbon greatly enhances its ammonia carrying capacity and adsorption rate.
- The phenomena of agglomeration and swelling as depicted by literature survey was completely overcome and heat and mass transfer was efficiently enhanced.
- Because no binder was used in the blocks so blocks were made under relatively high compression using UTM.

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