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Helium Purification by Gas Adsorption Method using Coconut Shell Activated Charcoal

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Abstract- **Helium stands out to be indispensable in the frontline research establishments and nuclear reactors of India. As superconducting technology requires liquid helium produced by helium liquefier, so Grade "A" helium is required as raw material for the liquefier.**

Recycle of helium gas is possible due to helium purifier which is the essential equipment of any major cryogenic laboratory. The helium purification system is based on cryosorption process at liquid nitrogen temperature and high pressure of 120 bar. This helium purification system purifies 60% pure helium to Grade "A" helium (99.995% minimum) i.e. the system reduces 40% air and moisture impurity to less than 50 ppm. The major components of this system are reciprocating compressor, adsorption column, helium purifier liquid nitrogen vessel, cryogenic heat exchangers, gas bag, stainless steel tubing, high pressure gas manifold system and instrumentation.
This report details the various stages of helium purification process, operating principle, utilization of indigenous products in cryogenic technology. Commissioning and testing of entire purification process which is done by taking 95 % pure helium and 5 % nitrogen has been presented in this document.

Key Words: Grade 4.5 Helium, Activated Charcoal LN2 vessel

1. INTRODUCTION

Helium, because of its extraordinary properties, stands out to be indispensable in frontier technologies especially in the domain of space, atomic energy, defence and medical sciences. The single largest application of pure helium is in cooling superconducting magnets, key to high energy accelerators, superconducting cyclotron and Magnetic Resonance Imaging (MRI) scanners. Also the low liquefaction temperature of pure helium makes it suitable for purging and pressurizing liquid hydrogen rocket propulsion system of cryogenic engine of space vehicles. Helium is an expensive and imported consumable. Therefore, helium purifier is an integral part of any cryogenic establishment to conserve the helium gas.

2. PURPOSE OF HELIUM PURIFIER

In a large cryogenic installation, generally helium is maintained in closed loop cycle. But, due to failure of the system or any of its components, the cold gas or liquid helium of the system expands to large volume and causes pressurization, resulting in blow off of safety relief valve. Because of the high cost and limited availability, helium is recovered in gas bag which causes ingression of air and moisture contaminants by diffusion. Similarly, laboratory experiments with cryostat and superconducting magnet, generates helium gas which is collected in gas bag. Recovery compressor compresses impure gas in gas manifold and results in oil contamination in high pressure storage. All these impurities are required to be eliminated to obtain Grade 4.5 helium gas suitable for running a liquefier. Hence, helium purifier is an indispensable device in any cryogenic set up.

3. OBJECTIVES OF THE PAPER

At present Indian academic institutions like IIT, NIT and research centers like Atomic Energy Establishment, Space Research Center, etc. import helium purifier which is not only very expensive in terms of foreign exchange but also a time consuming purchase affair. And also, we are to depend on foreign nations for its spares. Hence, National Institute of Technology, Rourkela has taken up the project of development of cryosorption based helium purifier in collaboration with Variable Energy Cyclotron Centre, Kolkata, funded by Board of Research in Nuclear Science, Mumbai, which primarily aims at developing helium purifier indigenously and to study short term and long term performance of system. The list of objectives of this project is as follows [24]:

(i) Design, development, installation and commissioning of cryosorption based helium purifier and its performance analysis with various concentrations of impurity and various types of adsorbents[24].
(ii) Proper documentation for future construction on commercial basis and technology transfer.

4. TECHNICAL SPECIFICATION OF HELIUM PURIFIER

Table No.1: Specification of Helium Purifier

rate	21 Nm^3/hr
Operating pressure and Temperature	120 bar (gauge) & 77K
Input gas purity	90% i.e. 10% air impurity
Output gas purity	99.995% (minimum) i.e. Grade 4.5 i.e. max. allowable impurity is 50 ppm
Run time for purification	6 (six) hours
Adsorbent	Coconut shell granular activated charcoal

5. DESCRIPTION OF BASIC PURIFICATION PROCESS

The basic principles involved in purification process are condensation of air impurities at heat exchangers and adsorption at high pressure and low temperature at activated charcoal bed [15]. The purifier system has been designed to purify 90% pure helium (10% air impurity) to 99.995% purity. Compressor sucks gas from helium gas bag and compress to 150 bar at flow rate of 21 Nm^3/hr. The gas is then passed through oil and water separator vessel and three heat exchangers in series. The first heat exchanger acts as dryer and the second and third one condense air and reduce the impurity in helium gas up to 0.84% only. Liquid air vessel then collects the condensed air, which is purged at regular interval. After that, a snow filter filters any ice coming with the impure stream. Now the impure helium gas, containing only 0.84% air impurity, enters the activated charcoal bed immersed in LN_2. The adsorber bed reduces air impurity to less than 50 ppm and thus yields Grade 4.5 helium. Back pressure regulator at the outlet maintains the operating pressure of 120 bar (g) at adsorber bed [12]. The purifier works in two phases – purification and regeneration [23].

6. DESIGN AND DEVELOPMENT OF THE PRINCIPAL COMPONENTS OF PURIFIER [24]

The helium purifier is very compact and complicated system. It has been made compact to reduce LN_2 consumption and to perform operation by single operator. The principal components of purifier system are 1) Compressor 2) Purifier LN_2 vessel with top flange 3) Adsorber columns 4) Tubular heaters 5) Gas bag 6) Pure and impure helium cylinder manifold 7) Piping network 8) Oil and moisture separator vessel 9) Liquid air separator vessel 10) Heat Exchangers

6.1 LN_2 vessel of Purifier and its Cover Flange

Super insulated LN_2 vessel houses all the cryogenic components like heat exchangers, adsorber bed, liquid air separator vessel, heaters and LN_2 level indicator. Inner diameter of vessel is 600 mm, inside depth is 1600 mm and annular space gap is 25 - 40 mm with 40 layers of multi-layer insulation. All the cryogenic equipments are resting within a suspended cage bolted to the bottom surface of top flange. The tubes interconnecting the inner and outer portions of purifier vessel are all passing through the top flange. This vessel has been designed and fabricated indigenously, the only imported component being the super insulation. Total LN_2 boil-off for 6 hours steady state purification run is 93.19 liters.

Ports are oriented on the top cover flange in such a way that the connecting tube lengths are minimum and aesthetic. All internal cryogenic components as well as tubular heaters are hanging from this flange and also the valve panel rests on this. Fig below shows assembled view of LN2 vessel with top flange.

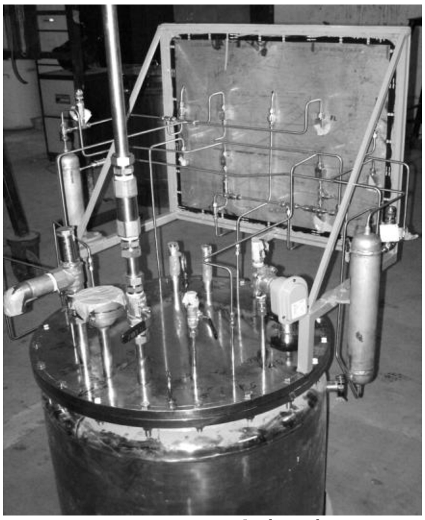

Fig. LN_2 vessel of purifier

6.2 Development of Adsorber Bed

Fig: U-shaped adsorber bed

The adsorbents commonly available in Indian market are Activated Alumina, Molecular Sieve or Zeolite, Silica Gel and Activated Charcoal. Nitrogen adsorption isotherm of coconut shell activated charcoal is better than other adsorbents and India is one of the major producers and exporters of coconut shell charcoal in the world [22].

Theoretical calculation reveals that after heat exchangers and phase separator, impure helium is saturated with 0.83 % air which will have to be adsorbed by activated charcoal. As per BET (Brunauer, Emmett and Teller 1938) equation [1], relationship followed for multi-layered adsorption, and considering 70% adsorption bed saturation, ***total charcoal*** *requirement is 7.82 Kg*.

Adsorber column dimension is determined based on [14] approach velocity of gas within adsorber and pressure of 120 bar (g). 50NB Sch80 ASTM A312 TP304L pipe is selected in which gas velocity is 0.02 meter/sec which is well within optimum range[24].

Adsorber bed of purifier consists of five numbers of U-shaped vertical stainless steel columns connected in series. The columns contain coconut shell activated charcoal of Indian origin. These are fully immersed in liquid nitrogen and hence maintaining temperature of around 77 K. Back pressure regulator maintains constant operating pressure of 120 bar (gauge) at adsorption bed.

Table No.2: Technical data of charcoal

Characteristics	Property of Charcoal
Grade	AC 4/8
Particle type	Granular
Sieve size	-4 +8 BSS
BET Surface area,m^2/gm,(min.)	1600
Moisture, percent by mass (min.)	4.5
Ash,percent by mass (max.)	4.0
Adsorption capacity in terms of Iodine number (min.), mg/gm	608

6.2 Development of Regeneration Process

Regeneration procedure of adsorbent bed is of two general types [11]: Pressure Swing Cycle and Thermal Swing Cycle. Thermal swing regeneration technique has been adopted for this project. Here activated charcoal is heated to 120^o C for desorption of gases for 4 hours and evacuated with rotary pump for last 2 hours up to the pressure of 10^{-3} mbar. The system is then purged with pure Grade 4.5 helium by reverse flow. The adsorber columns will be heated by six tubular heaters of 1 KW each positioned in LN_2 purifier vessel.

7. COMMISSIONING AND TESTING OF PURIFIER

After the completion of assembly of purifier, the pneumatic leak test was performed with nitrogen gas at test pressure of 165 bar(g) in accordance with ASME B 31.3[21].And for testing pure helium of 99.995% and 5% dry nitrogen contaminants are delivered to compressor from gas bag at atmospheric pressure. Compressor raises the pressure of impure helium to 120 bar and then supplied to purifier vessel and at the output four samples are taken which is checked off-line in Linde Multi-Component Detector.

8. RESULTS OF EXPERIMENTATION

The purity of output helium was checked in LMCD at VECC Kolkata and the final result we got the impurities, H2O, N_2, O_2 in vpm .The analysis of four samples of purified helium is presented in table 1:

Table No.3: Results of the four samples

Sl No.	Sample No.	H_2O (vpm)	N_2O (vpm)	O_2 (vpm)
1	1	1.70	2.10	0.20
2	2	2.30	1.50	0.10
3	3	1.40	1.40	0.30
4	4	1.50	2.80	0.30

The results show that, in all the samples total impurity, consisting of moisture, nitrogen and oxygen is less than 5 vpm, which means purified helium is better than Grade 4.5 or 99.995% helium. So, Grade 4.5 helium has been obtained from 5% nitrogen impurity in input helium for 3 hours nonstop run.

9. CONCLUSION

This work is a tiny contribution in the larger world of cryogenic technology. Indigenous development of helium purifier is not only quite cheaper but also gives a quantum jump in cryo-technology in our nation. Summarizing, the following may be seen as the significant contributions of the present investigation.

- Performance of helium purifier with various concentrations of impurities gives grade 4.5 helium.
- Coconut shell activated charcoal has worked fine as those being virgin, but we need to observe its performance in long run if purifier is run regularly for one year.
- Performance analysis of heat exchanger has been done.
- This purifier can be used commercially for purifying helium upto 40% impurity

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