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Experimental Investigation of a Cryogenic Filter for Separating Solid Carbon Dioxide Particles from Liquid Nitrogen

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Abstract: This paper presents an investigation of a new method of purifying cryogenic liquid using sintered metallic wire-mesh filter, which has the advantages of high purifying efficiency and preferred strength at absolutely low temperature. Experiments are conducted to purify solid CO_2 particles from liquid nitrogen. Temperature and pressure in the upstream and downstream of the filter, and the flow rate of carbon dioxide (CO_2) gas and liquid nitrogen are measured, with the gas content of filtrate analyzed using a CO_2 concentration detector. It is illustrated that after filtration, the purity of liquid nitrogen (volume fraction) is higher than 99.99%, which means that the volume fraction of CO_2 is less than 0.01%. Effects of operation parameters on the performance of the filter, such as pressure drop Δp and filtration efficiency E are analyzed quantitatively. The present conclusions will provide a guideline to the optimumal design and operation of sintered metallic wire-mesh filter in cryogenic application.

Key words: metallic wire-mesh filter, cryogenic liquid, pressure drop, filtration efficiency
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Introduction

Cryogenic liquids of high purity, such as nitrogen, helium and argon are preferred for a variety of industrial applications and aerospace research. There are several traditional methods to gain pure liquefied gas, such as simplified condensation and evaporation, rectification, pressure swing adsorption and membrane separation. In this paper, a new separation technology—filtration is put forward, which has some advantages over these traditional ones such as low energy cost, simplified system, long lifetime, high purifying efficiency and preferred strength at absolutely low temperature. The method is especially suitable for aerospace applications.

Sintered metallic wire-mesh filter, which owns small pores with diameter up to μm degree, is an excellent filtration medium. Theories about filtration mechanisms[1], stress analysis[2], influencing factors[3], and many results about its application at normal or high temperature have been reported, such as aerosol, talcum powder suspension and diesel emitted pollutants filtration. Recently, Richard[4] invented a filtration apparatus with sintered multi-channel ceramic tube to produce sterile cryogenic liquid, and successfully separated particles bigger than 0.2 μm. Takashi *et al*[5] used a three-layered filter made of low-humidity ceramic, zirconium oxide and quartz, with average pore size of 0.05 μm to 1.0 μm to get purified nitrogen. However, few literatures focused on the performance of sintered metallic wire-mesh filter at cryogenic temperature.

In this paper, a sintered metallic wire-mesh filter and the test rig are designed and built up, to investigate the performance of separating solid CO_2 particles from liquid nitrogen. Two important performance parameters of the filter, namely the pressure drop Δp and the filtration efficiency E are analyzed quantitatively at various operation conditions with different flow rate of feed slurry and volume fraction of CO_2 particles.

1 Experimental Facility

The filter core is made up of two layers of wire mesh sintered together to form an integrated porous element. The inner mesh is of very fine gauge and determines filtration accuracy (particle size). It is overlaid with coarse support mesh layers and protective outer mesh layers. The structural sketch of the filter is schematically shown in Fig. 1. Four parts of the filter and their dimensions are listed respectively in Table 1. Nominal filtration degree of the filter is 0.5 μm.

As shown in Fig. 2 the experimental apparatus comprises main parts: a filter unit including the filter core and the housing; a liquid nitrogen delivery system including the double-layer cryogenic dewar with delivery valves and flexible metallic hose; a CO_2 gas filling system involving a gas cylinder and a pressure regulating

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valve; a mixing chamber; a gas content analysis system in the downstream; a regenerating system; a measuring system including gas flow rate meters, temperature gauges and pressure sensors; a cryogenic safety and vacuum system comprising a burst disc, a safety valve, a vacuum pump and a vacuum gauge.

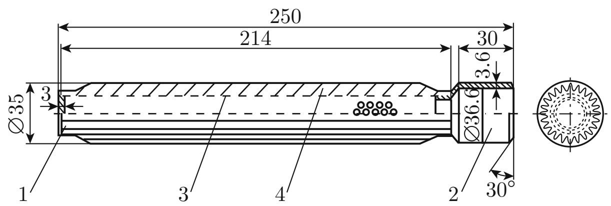
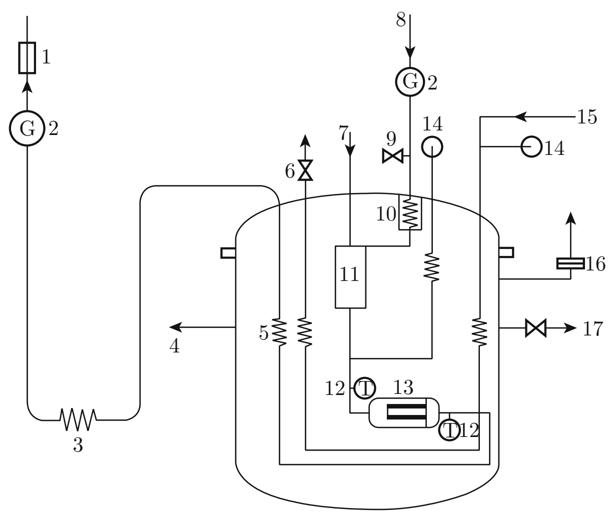


Fig. 1 Structural sketch of the sintered metallic wire-mesh filter (mm)

Table 1 Four parts of the metallic wire-mesh filter

Parts	Description	Dimensions /mm	Material
1	Cap	Ø22/19 × 3	AISI 316L
2	Boss	Ø36.6 × 3.6 × 42	AISI 316L
3	Perforated tube	Ø22 × 208 × 1	AISI 316L
4	Filter element	Ø35 × 214 × 3	AISI 316L



1—CO_2 concentration detector, 2—Gas flow rate meter, 3—Heat exchanger, 4—Vacuum gauge, 5—Spiral tube, 6—Safety valve, 7—Liquid nitrogen inlet, 8—CO_2 gas inlet, 9—Vent valve, 10—Pre-cooler, 11—Mixing chamber, 12—Temperature gauges, 13—Filter cell, 14—Pressure gauges, 15—Nitrogen gas inlet, 16—Burst disc, 27—Pump valve

Fig. 2 Schematic diagram of the test rig

The wire-mesh filter is placed in the housing. Before filtration, the test rig is evacuated from the pumping valve until the vacuum degree reached 50 mPa, to guarantee the heat insulation of dewar and pipelines. After all devices are connected and evacuation is completed, the leak rate of the rig is measured with a helium mass spectrometer leak detector, which should not exceed 1×10^{-10} Pa$\cdot$m^3/s. Then nitrogen gas is used to purge the pipelines, to clean residual air and water.

During filtration, CO_2 gas, the main impurities, is filled from the cylinder under a certain pressure into the pre-cooler which is full of liquid nitrogen at 77 K. Then cooled CO_2 and liquid nitrogen are mixed uniformly and thoroughly in a specially designed mixing chamber, to obtain CO_2 solid particles of evenly distributed size. The mixture enters into the housing, flowed through the filter, and CO_2 solid particles are deposited on the surface of the filter to form a filter cake because of the bigger particle size compared with the pore size of the filter and other filtration mechanisms such as gravity sedimentation, interception, diffusion, inertial impaction and so on. The filtrate comprising purified liquid nitrogen at the downstream of the filter is then heated by an air heat exchanger at room environment to atmospheric temperature. Gas content is analyzed using the CO_2 concentration detector. Mass of the cake grows until all the cells are deposited, then the filter should be back blown and regenerated with nitrogen gas at normal temperature.

2 Results and Discussions

2.1 Pressure Drop and Filtration Efficiency at Certain Operating Condition

Pressure drop and filtration efficiency are two primary performance indicators of a filter medium. Pressure drop across the filter medium is a measurement of its resistance to the flow through it. When the pressure drop reaches a preset value, the maximum allowable level, the cleaning cycle is initiated. Filtration efficiency defines how well the filter will remove contaminants, and it tends to build over time as particulates are collected, i.e., the efficiency gets higher as the filter gets dirty. Efficiency can be calculated as follow:

$$E = 1 - \varphi_{\mathrm{down}}/\varphi_{\mathrm{up}}, \tag{1}$$

where, φ_{down} represents the volume fraction of solid CO_2 in the filtrate, and can be measured with the CO_2 concentration detector; φ_{up} denotes the volume fraction of solid CO_2 in the feed slurry, and can be calculated according to the flow rate of CO_2 and nitrogen in the filter upstream.

To investigate the pressure drop and filtration efficiency evolution during filtration, flow rate of liquid nitrogen is controlled to be about 4.15 L/s (measured at the heat exchanger outlet, gas flow), which is enough to cool the pipelines and the filter to the freezing point of CO_2. At the same time, volume fraction of CO_2 is maintained to be 0.95%. Inlet pressure of filter is about 0.1 MPa.

Figures 3 and 4 show the change of pressure drop and filtration efficiency with time. It can be found that at the initial stage of filtration, pressure drop and filtration efficiency do not fluctuate strongly. That is because CO_2 particles deposited but has not form any consistent particle layer, and the main mechanisms are interception and diffusion. Hereafter, the pressure drop and filtration efficiency increased apparently and almost proportionally. The reason is that as the filtration process goes on, particle layer is formed locally because of the unevenly distributed fluid mixture in filter cell, which is inclined to flow through bigger pores in the metallic wire-mesh filter medium, on the other hand, CO_2 particles deposited on the area without any cake. With the spread of the layer surface, pressure drop increased sharply, but filtration efficiency maintained more than 99.99%. That is because full filter cake is formed on the surface, which plays the role of filter medium together with the wire-mesh, and filtration efficiency reached the maximum. At that time, all pores of wire-mesh are blocked, the filter needed to be back washed.

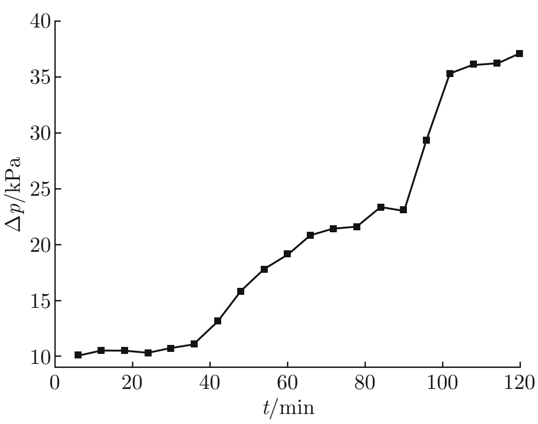


Fig. 3 Plot of pressure drop versus aging time

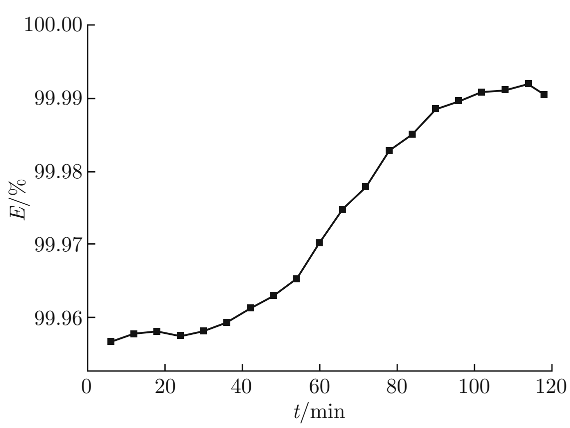


Fig. 4 Plot of filtration efficiency versus aging time

Compared with high temperature filtration, the change tendency of pressure drop and filtration efficiency are almost the same during the proportional phase. But some researches[6] demonstrated that the sharp increase of pressure drop is caused by the compression of the filter cake. So the compression at cryogenic temperature still needs to be studied.

2.2 Effect of Total Flow Rate of Mixture on Pressure Drop and Filtration Efficiency

Experiments are carried out with three different kinds of flow rates of mixture, 3.2, 4.7 and 5.2 L/s, respectively, to investigate the effects on pressure drop and filtration efficiency during filtration. Inlet pressure is controlled to be about 7.5 kPa, and the volume fraction of CO_2 is maintained to be 0.95%.

Figures 5 and 6 show the change of pressure drop and filtration efficiency at different flow rates of mixture. It can be found that the pressure drop fluctuates more strongly at higher flow rate, and the efficiency is also much higher, which means that almost all particles are trapped by the filter and the purity of nitrogen (volume fraction) reached more than 99.99%. As far as lower flow rate, the pressure drop maintained proportionally until 120 min, and the efficiency increased continuously to be more than 99.99%.

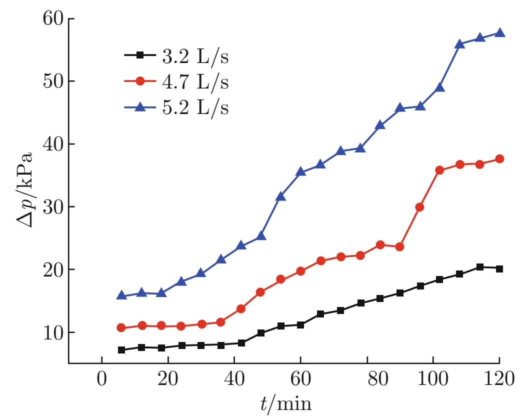


Fig. 5 Relation of pressure drop with different flow rate of mixture

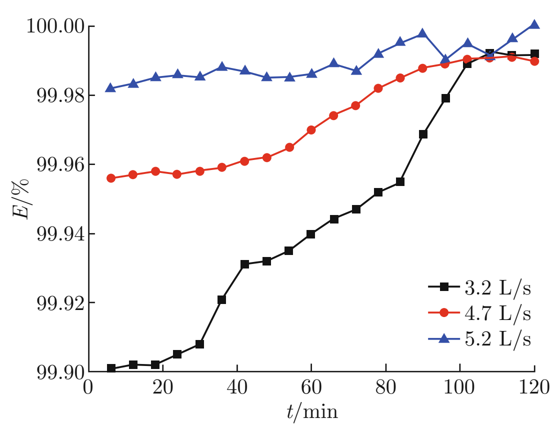


Fig. 6 Relation of filtration efficiency with different flow rate of mixture

Change of pressure drop is similar to the trend at high temperature, which means to some degree that the filtration mechanisms at high temperature are applicable to cryogenic filtration. However, the efficiency is much higher than that at cryogenic temperature, which is maybe caused by other further mechanism such as particle sticking under low temperature[7].

2.3 Effect of Volume Fraction of CO_2 on Pressure Drop and Filtration Efficiency

Experiments are carried out with three different volume fractions of CO_2, 0.95%, 1.95% and 2.95%, respectively, to investigate the effect on pressure drop and filtration efficiency during filtration. Inlet pressure is controlled to be about 7.5 kPa, and flow rate of liquid nitrogen is controlled to be about 4.15 L/s.

Figures 7 and 8 show the change of pressure drop and filtration efficiency with volume fraction of CO_2. It can be found that at a specified flow rate of liquid nitrogen, pressure drop increases more quickly and the filter is more efficient at the condition with higher CO_2 volume fraction. The reason is that, the filter pores are more likely to be blocked under high particle concentration, and the filter cake is easier to be formed on the surface.

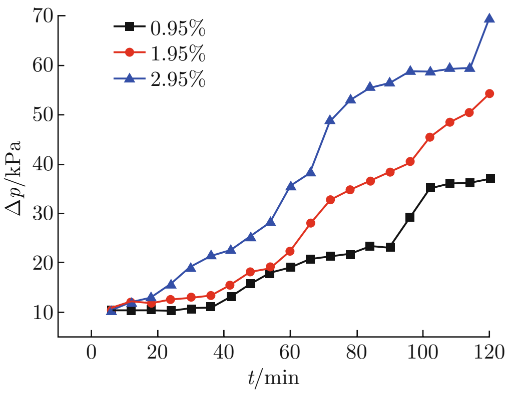


Fig. 7 Relation of pressure drop with different volume fraction of CO_2

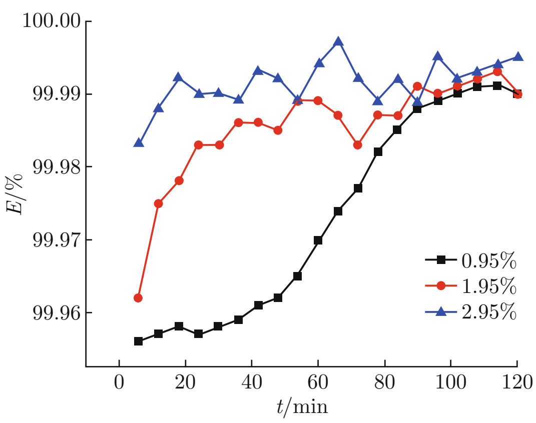


Fig. 8 Relation of filtration efficiency with different volume fraction of CO_2

Compared with high temperature filtration, increase trend of pressure drop here is more coincident with exponential distribution, and the ones at high temperature with logarithmic distribution[8]. That is because some CO_2 particles may be melted while adjusting the flow rate of liquid nitrogen in part at the first stage of filtration, leading to slower increase speed of pressure drop.

3 Conclusion

(1) The sintered metallic wire-mesh filter is a highly efficient type with low pressure drop at low temperature. Almost all CO_2 particles are trapped, cleaned liquid nitrogen with purity (volume fraction) of higher than 99.99% is obtained.

(2) The pressure drop and filtration efficiency increase with increasing flow rate of the mixture and the volume fraction of CO_2. Particle layer and filter cake are formed and play main roles of filter medium together with the metallic filter.

(3) Evolution trend of pressure drop and filtration efficiency at cryogenic temperature are different more or less from that at normal or high temperature, because of the compression of the filter cake, filtration mechanism such as particle sticking and so on.

Efforts will be made to research the mechanisms at cryogenic temperature, and the back washing performance of the sintered metallic wire-mesh filter.

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