Pergamon

Talanta, Vol. 41, No. 2, pp. 269–272, 1994

0039-9140/94 $6.00 + 0.00

PREPARATION OF PERLITE-BASED CARBON DIOXIDE ABSORBENT

HUANNAN HE, LIXIN WU,* JIANYING ZHU and BOHUA YU
Shanghai Institute of Metallurgy, Chinese Academy of Sciences, Shanghai, 200050, People's Republic of China

(*Received* 1 *March* 1993. *Revised* 9 *July* 1993. *Accepted* 12 *July* 1993)

Summary—A new highly efficient carbon dioxide absorbent consisting of sodium hydroxide, expanded perlite and acid–base indicator was prepared. The absorption efficiency, absorption capacity, flow resistance and color indication for the absorbent were tested and compared with some commercial products. The absorbent can reduce the carbon dioxide content in gases to 3.3 ppb (v/v) and absorbs not less than 35% of its weight of carbon dioxide. Besides its large capacity and sharp color indication, the absorbent has an outstanding advantage of small flow resistance in comparison with other commercial carbon dioxide absorbents. Applications in gas analysis and purification were also investigated.

Carbon dioxide absorbents are widely used not only in analysis and purification of gases but also in the chemical and food industries, clinics, spacecraft and submarines, *etc.* There have been many carbon dioxide absorbents which can be divided mainly into liquid absorbents such as soda, ammonia, ethanolamine,[1] and solid absorbents such as calcium oxide, soda lime, soda asbestos, *etc.* Alkali metal hydroxide or carbonate impregnated in porous carriers, such as active carbon,[2] porous alumina,[3] polytetrafluoroethylene[4] are most popular. However, a few carbon dioxide absorbents can be used in gas analysis and purification. In the past few decades soda asbestos has become the most favorable carbon dioxide absorbent for use in gas analysis. The earlier soda asbestos easily formed a thin film of carbonate on its surface, which reduced the absorption[5] and produced a large flow resistance.[6] After being improved, the commercial soda asbestos available today is satisfactory in absorption efficiency, capacity, *etc.* but the problem of flow resistance is not yet solved. Not only will the change of flow resistance cause a change of gas flow-rate but also will greatly decrease the practical utilization ratio.

Expanded perlite used as supports has been reported in several patents.[7–9] The authors have prepared a perlite-based magnesium perchlorate desiccant which is superior to anhydrous magnesium perchlorate in performance.[10] Again we aim to use expanded perlite instead of asbestos to carry sodium hydroxide. Thus a new carbon dioxide absorbent called soda-perlite, in which sodium hydroxide is impregnated in expanded perlite, has been prepared. The performance tests show that the soda-perlite has advantages over soda-asbestos in absorption efficiency, especially in flow resistance.

EXPERIMENTAL

Preparation

Expanded perlite samples are floated on water and dried at 120–180°C, then sieved. A particle size passing through 10-mesh and retained on a 20-mesh screen is used as carrier-base material; 200 g of C.P. grade sodium hydroxide and 0.15 g of phenolphthalein are added into 85 ml water. When dissolved completely the solution is mixed with 90 g of the refined perlite homogeneously. The perlite-based semi-product is dehydrated at a temperature of 130–140°C in a vacuum electric furnace for 2–6 hr. After cooling, the final product named HP-1 CO_2 absorbent is obtained.

Performance tests

Absorption efficiency. The absorption efficiency can be expressed in the residual (unabsorbed) carbon dioxide amount (ppb, v/v) of a gas mixture that passed through carbon dioxide absorbents. An HP-5880A gas chromatograph (Hewlett Packard Co., U.S.A.) equipped with

---

*Author for correspondence.

FID was used. The set up for the measurement was presented in Ref. 11.

Absorption capacity. Absorption capacity for carbon dioxide is expressed as a percentage of the carbon dioxide weight absorbed in per unit weight of the absorbent. A gas mixture (6.7% CO_2 in nitrogen) was continually passed through a glass tube (15 mm I.D. × 100 mm) packed with carbon dioxide absorbent at a flow-rate of 200 ml/min until the absorbent was exhausted.

Flow resistance. A gas mixture (2.1% CO_2 in nitrogen) was sent through a tube (15 mm I.D. × 100 mm) packed with absorbents. The measurement method is similar to Ref. 10.

RESULTS AND DISCUSSION

Quality of perlite

Like other materials used as carriers, expanded perlite is used because of its large surface area and good mechanical strength. Its chemical structure and composition are influential in its load of sodium hydroxide. Although zeolitization of perlite in sodium hydroxide solution under particular conditions has been reported,[12,13] which indicates its chemically unstable properly in sodium hydroxide, the perlite samples used in our experiments did not show its reaction with sodium hydroxide. The reason cannot just be attributable to their different chemical compositions due to their different source locations. An important reason, we think, is their different preparation conditions. It was found that expanded perlite with a lower SiO_2/Al_2O_3 ratio is preferably used to carry sodium hydroxide and lower water content in the product is favorable to its stability. Particle size of expanded perlite is not critical, preferably 10–20 mesh.

Ratio of NaOH/perlite

The weight ratio of sodium hydroxide to expanded perlite is preferably approximately 2:1. Low ratio will reduce the absorption capacity, whereas high ratio will easily make the product agglomerate due to the limited carrying capacity of expanded perlite, and as a result of this the absorption capacity will decrease.

Dehydration

In investigation of long stability, preliminary experiments have shown that the absorption capacity reduced to 12% from the original 32% after 3 months if the product is not dehydrated. When dehydrated, however, the product became very stable and the absorption capacity is raised. The absorption capacity is almost unchanged, roughly 35%, after one year. Although dehydration is important for the product its condition is not strict. Temperature between 120 and 150°C and a low vacuum are enough for dehydration.

Color indication

Phenolphthalein is used as indicator in the absorbent. Compared with other acid–base indicators tested, phenolphthalein is very cheap and sharp in color change. The absorbent can turn to rose-red from original pale purple after absorption of carbon dioxide.

Absorption efficiency

The results for measurement of absorption efficiency of carbon dioxide absorbents including some other commercial products are listed in Table 1. It can be seen from Table 1 that the carbon dioxide residue passed through HP-1 CO_2 absorbent and soda asbestos (E. Merck Co.) are the lowest. Effects of temperature, flow velocity, carbon dioxide concentration of gas

Table 1. Absorption efficiency of carbon dioxide absorbents

Absorbents	Number of determinations	Average of CO_2 residues (*ppb* (*v*/*v*))	Relative standard deviation (%)
HP-1 CO_2 absorbent (our laboratory)	6	3.3	3.0
Soda asbestos (E. Merck Co., Germany)	3	3.5	4.4
Ascarite II (LECO Co., USA)	4	11.9	4.4
Carbosorb AS (B.D.H. Co., UK)	4	7.2	3.3
Soda lime (Shanghai Reagent Factory, China)	4	365	3.1

Table 2. Absorption capacity of carbon dioxide absorbents

Absorbents	HP-1 CO_2 absorbent	Soda asbestos (E. Merck Co.)	Ascarite II (LECO Co.)	Carbosorb AS (B.D.H. Co.)	Soda lime (Shanghai)
Average of capacity (%)	35	28	24	34	24

mixtures on absorption efficiency were also investigated. The detailed results have been given in Ref. 11.

Absorption capacity

With the measurement described in the Experimental Section the absorption capacities of HP-1 CO_2 absorbent and other commercial products are listed in Table 2. It is seen that HP-1 CO_2 absorbent has the largest capacity. It should be pointed out that the packing height of Carbosorb AS in the absorbing tube is only 20 mm (others are 50 mm) its large flow resistance makes it impossible to complete the absorption of carbon dioxide if packed in 50 mm in height. The capacity of Carbosorb AS can rarely reach 34% in practice.

Flow resistance

It can be seen from Fig. 1 that all of the absorbents have similar curves for flow resistances, the value for HP-1 CO_2 absorbent is the smallest. In Fig. 2, however, their change curves are very different. The flow resistance of soda asbestos type absorbents, especially for Carbosorb AS, quickly increase with the absorption of carbon dioxide. For instance, when Carbosorb AS has spent 10 mm in height the flow resistance is roughly 820 mmHg at the flow-rate of 0.5 l./min. Such large flow resistance makes it difficult to continue working in practice since the gas line cannot ordinarily bear such high pressure. Contrasting sharply, HP-1 CO_2 absorbent not only has a very small flow resistance but also has a small flow resistance change after being absorbed completely by carbon dioxide.

Application

HP-1 carbon dioxide absorbent can be widely used in analysis and purification of gases, such as in determination of carbon in metals by combustion in oxygen, purification of carrier gases, and preparation of high pure gases. The absorbent has been used to determine carbon in metal reference samples by the combustion-gravimetric method. The analytical results in Table 3 show that the absorbent has a high absorption efficiency for carbon dioxide. It has also been used in the instrument for gas analysis, such as infrared carbon/sulfur analyzer. HP-1 CO_2 absorbent has the advantage of small flow resistance and sharp color change in comparison with other commercial products, which is convenient in controlling a steady flow-rate and identifying the efficiency of absorbent. The absorbent is more efficient than moelcular sieves in absorbing micro carbon dioxide for use in purification of high pure gases.

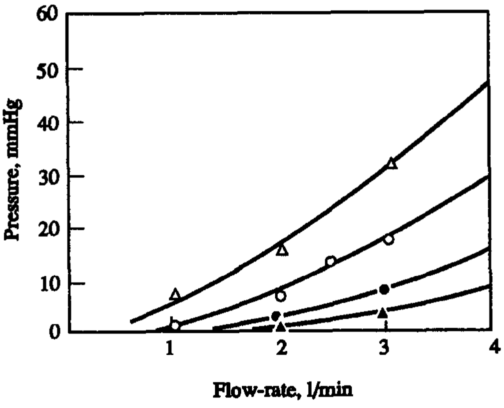

Fig. 1. Relationship between flow resistance and gas flow-rate. △ Carbosorb AS (B.D.H. Co.), ● Soda asbestos (E. Merck Co.), ○ Ascarite II (Leco Co.), ▲ HP-1 CO_2 absorbent (our laboratory).

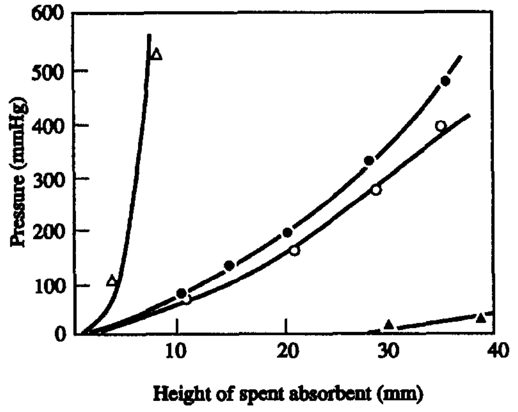

Fig. 2. Relationship between flow resistance and height of spent absorbents. △ Carbosorb AS (B.D.H. Co.), ● Soda asbestos (E. Merck Co.), ○ Ascarite II (Leco Co.), ▲ HP-1 CO_2 absorbent (our laboratory), flow-rate 500 ml/min.

Table 3. Results of measurements for carbon in metal reference samples by combustion-gravimetric method

Reference samples	Average of determination	Number of determinations	Relative standard deviation	Relative error with standard value
Cast iron, 2.48% C (Bureau of analysed Sample Ltd., UK)	2.48	3	0.0	0.0
Carbon steel, 0.34% C (Shanghai Inst. of Iron and Steel, China)	0.34	3	2.9	0.0
Ni–Cu–Cr, 3.06% C (Bureau of Analysed Sample Ltd., UK)	3.06	3	2.1	0.0
Carbon steel, 0.53% C (Shanghai Inst. of Iron and Steel, China)	0.54	3	1.1	1.8

CONCLUSION

A new carbon dioxide absorbent consisting of sodium hydroxide, expanded perlite and acid–basic indicator was prepared. The highly efficient absorbent named HP-1 carbon dioxide absorbent is superior to some commercial CO_2 absorbents in performance. The CO_2 residue passed through HP-1 CO_2 absorbent can be lowered to 3.3 ppb (v/v), which is lower than any of the commercial products tested in our experiments. Besides large absorption capacity ($\geq 35\%$) and sharp color change, the new absorbent has an outstanding advantage of small flow resistance compared to other CO_2 absorbents. Application tests indicate that the absorbent is more convenient and efficient in gas analysis and purification.

REFERENCES

1. A. L. Kohl and F. C. Riesenfeld, *Gas Purification*. Gulf, Houston, 1979.
2. Toppan Printing Co., Ltd, *Japanese Patent* 83177137, 1983.
3. L. H. Slaugh and C. L. Willis, *US Patent* 4433981, 1984.
4. M. Katz and N. Conn, *US Patent* 3909206, 1974.
5. R. Wildenstein, *Mikrochemie ver. Mikrochim. Acta*, 1952, **39**, 194.
6. J. Koizumi, Y. Youchi and S. Kinoshita, *Bunseki Kagaku*, 1967, **16**, 42.
7. H. H. Houstoh, *US Patent* 2967153, 1961.
8. J. Siebel and H. Pape, *Ger. Offen.* 1260441, 1968.
9. W. Adolf, N. Karlheinz and L. Alois, *Ger. Offen.* 1946277, 1971.
10. L. X. Wu and H. N. He, *Talanta*, accepted.
11. L. X. Wu and H. N. He, *Talanta*, 1992, **39**, 1081.
12. P. L. Antonucci, M. L. Crisafulli, N. Giordano and N. Burriesci, *Mater. Lett.*, 1985, **3**, 302.
13. N. Burriesci, M. L. Crisafulli, N. Giordano, J. Bart and G. Polizzotti, *Zeolites*, 1984, **4**, 384.