

This article was downloaded by:

On: 25 January 2011

Access details: *Access Details: Free Access*

Publisher *Taylor & Francis*

Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



Separation Science and Technology

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713708471>

Particle Chromatography

Vincent H. S. Kuo^{ab}, William R. Wilcox^a

^a CHEMICAL ENGINEERING AND MATERIALS SCIENCE DEPARTMENTS, UNIVERSITY OF SOUTHERN CALIFORNIA, LOS ANGELES, CALIFORNIA ^b Allied Chemical Corporation, Solvay, New York

To cite this Article Kuo, Vincent H. S. and Wilcox, William R.(1973) 'Particle Chromatography', Separation Science and Technology, 8: 3, 375 — 377

To link to this Article: DOI: 10.1080/00372367308058010

URL: <http://dx.doi.org/10.1080/00372367308058010>

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: <http://www.informaworld.com/terms-and-conditions-of-access.pdf>

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

COMMUNICATION

Particle Chromatography

VINCENT H. S. KUO* and WILLIAM R. WILCOX

CHEMICAL ENGINEERING AND MATERIALS SCIENCE DEPARTMENTS
UNIVERSITY OF SOUTHERN CALIFORNIA
LOS ANGELES, CALIFORNIA 90007

Abstract

Particle mixtures can be separated into bands by slowly increasing the freezing rate during directional solidification. Continuous size classification of spherical particles can similarly be obtained.

We (1, 2) and others (3-9) have shown that a foreign particle is generally rejected during solidification unless the freezing rate exceeds a critical value V_c . Above V_c the particle is trapped and incorporated in the solid. Values for V_c are sensitive to the particle-melt combination. Although one cannot yet predict V_c for a particular pair, it is expected that factors such as relative thermal conductivity, melt viscosity, and surface properties of particle and solid all influence V_c . The critical freezing rate of spherical particles has been found to vary roughly with the reciprocal of particle diameter. The critical rate for rough particles is higher than for spherical particles and is less size dependent.

These previous results suggest that one might use this particle pushing phenomenon to separate particle mixtures by size and type. The idea is to disperse the particles in a relatively pure liquid which does not act as a solvent for the particles, and then directional-freeze the liquid at a

* Present address: Allied Chemical Corporation, Solvay, New York 13209.

steadily increasing rate—programmed solidification. As V_c is exceeded for each type of particle, it will be trapped in a band. Thus the name “particle chromatography.” Because of the specificity of V_c , it might also be possible to identify particles by determining V_c in several liquids.

The purpose of the experiments reported here was to test this concept of particle chromatography.

EXPERIMENTAL METHODS

Two experimental techniques were tried—the vertical Bridgman-Stockbarger technique and horizontal zone melting with rotation—as discussed in detail elsewhere (2). Carbon, copper, red ferric oxide, and silver particles were ultrasonically dispersed in molten naphthalene which was then poured into 10.5 mm i.d. Pyrex tubes. Particles were included only in material at the front end of the tube, in tubes intended for zone melting experiments, so that the initial zone contained all particles present. The remainder of the naphthalene contained no particles. All but the smallest particles of each type settled to rest on the interface during the Bridgman experiments. Particles were completely suspended in the rotating zone-melting experiments.

In a preliminary Bridgman experiment with ferric oxide and carbon particles, bubbles formed at the interface and caused irregular trapping of particles, preventing appreciable separation. Bubble formation was prevented in subsequent experiments by sealing the tube containing solidified naphthalene in a vacuum of 0.05 Torr prior to performing the experiments. Although bubbles were not a problem in horizontal zone melting, most of the gas was removed by a preliminary rapid zone pass of the naphthalene which initially contained no particles.

In order to avoid trapping at the wall, it was also found necessary to use a concave interface.

RESULTS

Carbon and copper were quantitatively separated by horizontal zone melting with rotation at 44 rpm, as shown in Fig. 1. The zone travel rate was increased from 25 to 75 mm/hr over 4 hr. Carbon particles began to be trapped at 34 mm/hr. The carbon was all trapped in the center of the ingot over a length of 2 cm. Copper was trapped uniformly across the ingot from 54 to 75 mm/hr.

Carbon and copper were also separated by the vertical Bridgman method, although the trapping order was reversed. By programming the

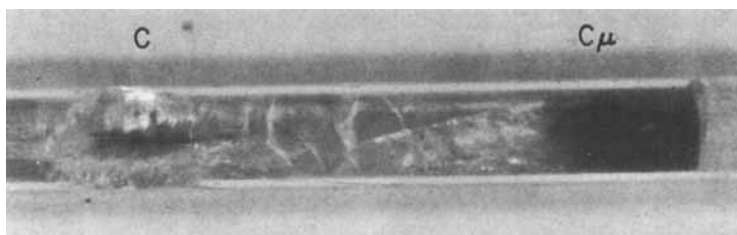


FIG. 1. Carbon and copper particles were separated during programmed solidification of naphthalene in horizontal zone-refining with rotation (HR-16 M).

tube lowering rate from 10 to 40 mm/hr, copper was trapped in the middle of the tube and carbon in the upper portion.

A limited amount of size classification was obtained for silver particles using the Bridgman-Stockbarger method by increasing the freezing rate from 6 to 26 mm/hr. The bottom contained predominantly large particles. The problems probably arose from the irregular shapes of the particles and the polycrystallinity of the solid naphthalene. Spherical particles with a single crystal solid should yield perfect continuous size classification even of submicron particles.

Acknowledgments

This research was supported by Grant No. 4528-AC6 from the Petroleum Research Fund (administered by the American Chemical Society) and by Grant No. DAHC15-72-G7 from the Advanced Research Projects Agency of the Department of Defense.

REFERENCES

1. V. H. S. Kuo and W. R. Wilcox, *Ind. Eng. Chem., Process Des. Develop.*, in press.
2. V. H. S. Kuo, "Removal and Separation of Particles by Crystallization," Ph.D. Dissertation, University of Southern California, Los Angeles, 1973.
3. C. W. Correns, *Discussions Faraday Soc.*, 5, 267 (1949).
4. A. E. Corte, *J. Geophys. Res.*, 67, 1085 (1962).
5. D. R. Uhlmann, B. Chalmers, and K. A. Jackson, *J. Appl. Phys.*, 35, 2896 (1964).
6. P. Hoekstra and R. D. Miller, *J. Colloid Interface Sci.*, 25, 166 (1967).
7. J. Cisse and G. F. Bolling, *J. Crystal Growth*, 11, 25 (1971).
8. J. Cisse and G. F. Bolling, *Ibid.*, 10, 67 (1971).
9. M. V. Pikunov, *Metalloved. i Obrabotka Tsvetnykh Metal. i Splavov*, Sbornik State, 1957, 55; through *Chem. Abstr.*, 52, 16826c (1958).

Received by editor December 12, 1972