PILOT SMALL-SCALE INDUSTRIAL PLANT FOR EXTRACTION AND SEPARATION OF FULLERENES

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INTRODUCTION

The fields for practical fullerene application are spread every day but the main barrier to the mass utilization of fullerene products is high cost of production. Methods and equipment described in literature for fullerene extraction from the fullerene containing soot and their following separation into fractions are expensive and low-output as they were oriented to prepare product under laboratory conditions in little amount necessary for conducting scientific investigations [1-4].

Therefore the pilot small-scale industrial plant has been designed to produce fullerenes in amount necessary for commercial projects. Its design is described below.

DESCRIPTION OF THE PLANT

The plant is designed to produce the concentrated solutions of C_{60} and C_{70} fullerenes from the fullerene containing soot and consists of extraction, separation and evaporation modules.

The extraction module is designed for extraction of the fullerene mixture from the fullerene containing soot by method of periodic extraction with organic solvents (toluene) in the fluidized bed. The production rate of the module is 0.6 kg of soot per shift. The solvent consumption is 35 l per shift.

The separation module provides separation of the prepared solution of the fullerene mixture (extract) into fractions. Separation is conducted in the chromatographic column. As sorbent МПГ-6 or Б-6 graphite having 0.1-0.26 mm fractions is used in it. 0.4 l of saturated solution of the fullerene mixture (~0.8 g of dry extract) may be injected into the column at a time. The fullerene yield from the column is controlled visually by the solution color or with photospectrometer. Toluene is used as eluent.

The separation cycle lasts 25-30 min. 12-15 l of toluene is spent per one cycle.

The columns are capable of processing to 50 g of dry extract without graphite replacement.

The evaporation module concentrates C_{70} solutions to the necessary concentration and provides extraction and separation modules with pure toluene what decreases considerably amount of pure toluene required for the technological process (to 3-4-fold consumption of toluene per one cycle of extraction and separation).

The evaporation process is conducted under vacuum. The heat required for the solution evaporation is fed from the electric heating element through the wall of the reboiler. The production rate of each evaporation module is ~6 l/hr.

Scheme of the plant with minimum necessary equipment is shown in Fig.1.

Each module in the plant can work both independently and parallel to each other. The system for blow-down of tanks with argon and the system for filling the service tanks with solutions and solvents are common for all modules.

Modules for extraction and separation are controlled manually. The evaporation module contains an automatic control system that provides maintenance and control over parameters in the technological process (temperature, pressure, level of the solution in the reboiler etc.), giving the warning signals and the emergency detachment of the module in extreme situations.

CONCLUSIONS

Due to the choice of comparatively efficient and productive method for extraction of fullerenes from the soot and their separation into fractions, and optimum technological parameters and construction design, the plant makes possible to produce the cheap fullerene products in rather large amount. Thus broad possibilities are opened for the applied use of fullerenes and widening the fields for their application.

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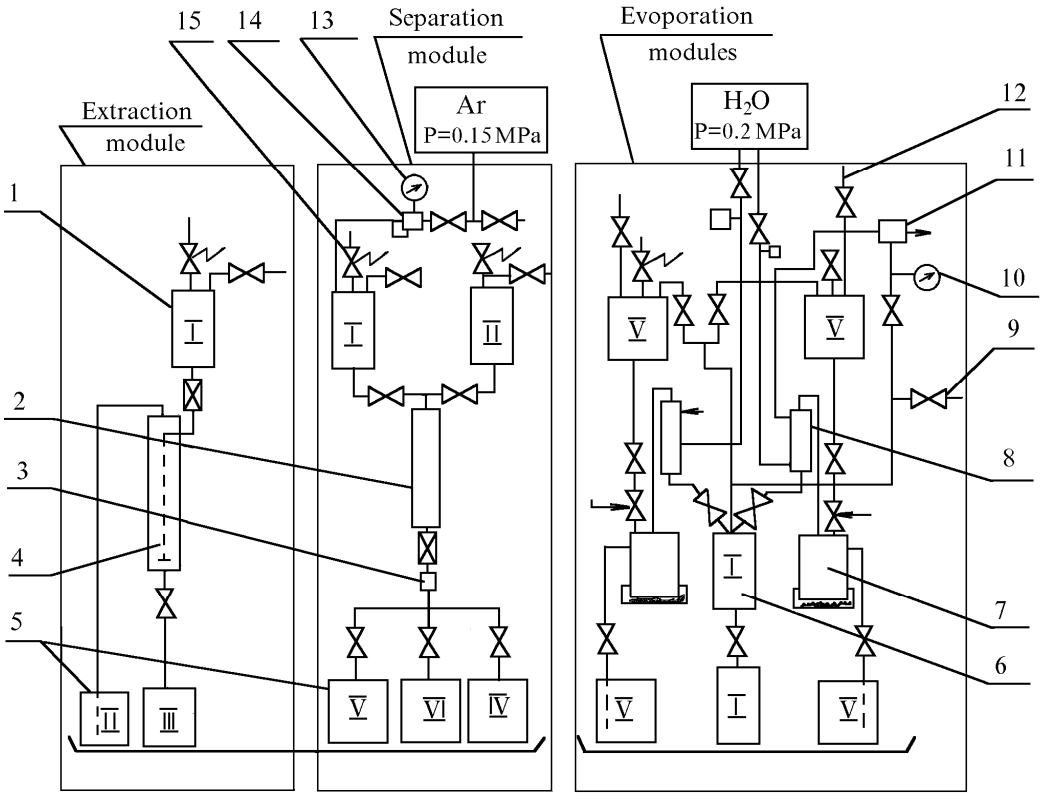

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Fig.1. Scheme of the pilot small-scale industrial plant for extraction and separation of fullerenes. I - solvent; II - extract; III - soot residue; IV - solution of C_{60} fullerenes; V - solution of C_{70} fullerenes; VI - solution of C_{60} and C_{70} fullerenes mixture. 1 - service tanks; 2 - chromatography column; 3 - cells; 4 - extraction column; 5 - tanks-collectors; 6 - tank-accumulator; 7 - reboiler; 8 - condenser; 9 - blow-down cock; 10 - vacuum-gauge; 11 - water jet pump; 12 - filling cocks of service tanks; 13 - manometer; 14 - reducer; 15 - safety valves.