A PROCEDURE FOR SUPPORT GRANULES POSSESSING HIGH MECHANICAL STRENGTH

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Introduction

The use of granular pesticides due to their numerous advantages has steadily increased all over the world [1]. Various technological methods of granulation have been developed to meet the growing demands.

In many cases the various pesticides or pesticide combinations are transformed into the granular form by applying support granules produced by appropriate procedures. The end product is obtained by impregnating the support granules with the solution, melt or suspension of the pesticide [2, 3].

The characteristics of granules prepared in the above-mentioned ways basically depend on the nature of the support granules. To manufacture favourable granular pesticides the support granules have to fulfil several requirements.

Above all, they must have great mechanical strength since usually they will be impregnated by spraying in a rotating drum. Strength is generally ensured by some kind of binder (e.g. a polymer) and can be increased by higher percentages of the binder, which will, however, reduce the adsorptive power and sorptive power of the support granules for the active agent, this being a great disadvantage for applications. Accordingly, great strength and relatively good sorptive power are both essential demands for support granules.

Translated into practice, this means that mechanical strength, above all, resistance to abrasion, should be increased without decrease of sorptive power, and as far as possible without changing the percentage of the polymer.

Theoretical discussion

It is known from the literature that water-soluble polyvinyl alcohol is transformed in the presence of alkali or mineral acids into a polymer insoluble or slightly soluble in water by intramolecular ether formation accompanied

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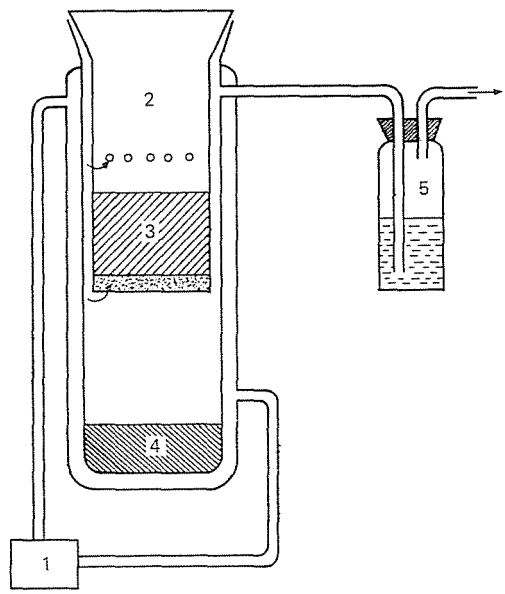
by the elimination of water. This fact suggested the idea to manufacture support granules by utilizing initially water-soluble polyvinyl alcohol and subsequently turning it partly or totally water-insoluble by etherification. The transformation of the binder was expected to improve the mechanical characteristics of the product, above all its resistance to abrasion.

Experimental

Support materials with various adsorption properties (bentonite, diatomite, brown coal powder) were used in the experiments. The finely powdered support materials were mixed with a 5% aqueous solution of polyvinyl alcohol (Mowiol 8—88, manufactured by Hoechst) in amounts to obtain, after drying, end products with a predetermined mass ratio of support material and polyvinyl alcohol.

The paste-like material was dried at 50 °C, crushed and screened. The fraction 0.2 mm $< d <$ 0.63 mm was used in further experiments.

Three sets of experiments were carried out, treating the granules in an atmosphere of HCl at 70, 80 and 90°, resp., with the variable within each set being the time of the treatment, namely 0, 30, 60 and 120 minutes.



1. thermostat
2. removable vessel with porous bottom
3. granules
4. conc. HCL
5. washing flask

Fig. 1. Apparatus for the thermal treatment of support granules

The apparatus used (Fig. 1) was thermostated (1) to the requested temperature. Concentrated HCl was then introduced into the lower part of the apparatus (4) subsequently a vessel with a porous bottom (2) containing the granules to be treated was placed into the apparatus and removed at the end of the experiment.

The most important mechanical property: resistance to abrasion of both thermally treated and untreated granules was tested by shaking 10 g of the material in a 25 cm^3 vessel for 8 hours on a shaking-table, screening through a 0.2 mm screen and weighing the material passing through the screen, that is, abraded during shaking on an analytical balance. The results were expressed as percentage of abrasion.

The sorptive power of the granules for the active agent was determined — in analogy to the so-called oil number determination [4] — by the volume of helianthus seed oil, in cm^3, sorbed by 100 g of granules.

Helianthus seed oil was added to 10 g of granules dried at 105 ± 2 °C drop by drop at intervals of 5 seconds, from a microburette as long as the granules, constantly stirred with a spatula, remained dry to touch and the granules did not stick together. The volume of oil spent, multiplied by ten, was termed the oil sorption of the sample. The data calculated were the means of three measurements.

Results

Our experiments demonstrated that when support granules prepared of materials with various characteristics and utilizing different quantities of polyvinyl alcohol as binder, are subsequently modified in hydrochloric acid

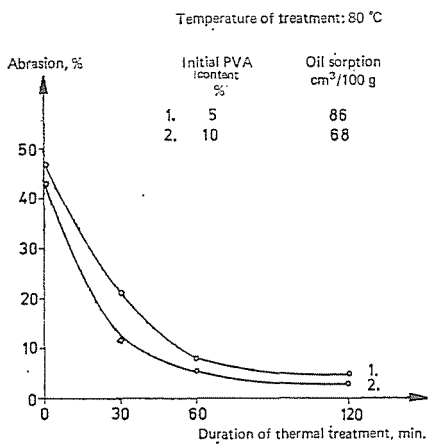


Fig. 2. Resistance to abrasion of support granules made of diatomite and polyvinyl alcohol binder *vs.* period of thermal treatment

at temperatures exceeding 50 °C by intramolecular etherification of the binder, the mechanical strength of the product obtained depends on the measure of modification (duration of the thermal treatment). The sorptive power for the active agent is not affected by the modification: deviations exceeding $\pm 5\%$ between the oil sorption values of thermally treated and untreated samples were never experienced. The results of two experiments carried out with granules made of diatomite and polyvinyl alcohol binder are presented by way of example in Fig. 2 demonstrating that the resistance to abrasion of the granules considerably increases by thermal treatment performed at 80 °C for various periods.

A patent was applied for the procedure described in this paper.

Summary

A procedure was developed for producing support granules for pesticides, distinguished by high mechanical strength and favourable sorptive power for active agents. The essence of this procedure is a subsequent modification of the granules by intramolecular etherification of the water-soluble polyvinyl alcohol used as binder.

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