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Post: DF/MEX/78/017/11-86/32. ssion Report: Flastics Films in Agricultural Applications (Mexico) Avrom I. Medalia

SIDMARY

The manufacture of black polyethylene film is divided into two stages, which are carried out by different companies: preparation of concentrates (masterbatches), and production of the film. Problems of a masterbatch producer arise from the limited grades of raw material available in Mexico, and from the uncertain level of quality control. Cther problems arise from the type of mixing equipment installed and from lack of suitable tests for product (masterbatch) quality.

<u>CIGA</u> is functioning as a national center for plastics technology and has on-going programs for agricultural plastics film. CIGA is generally well set up for polymer studies but is deficient in equipment and expertise for powdered and colloidal materials such as carbon black.

My chief contributions on this mission were (1) general lectures and advice on carbon black, mixing, and evaluation of dispersion; (2) introduction of the best method (tape extrusion) for carbon black dispersion in masterbatches, using equipment available at CIQA; (3) a visit (with a CIQA representative) to a masterbatch producer; and (4) recommendation and specific advice to CIQA to set up a program for testing of carbon black.

Recommendations are given for further action by CIQA under the headings of Technical, Programs, Equipment, and Organizational.

TECHNICAL PROBLEMS OF MEXICAN PLASTICS INDUSTRY re MISSION SUBJECT

Thin plastics (polyethylene) film is manufactured by the film blowing process. The film for agricultural applications may be either clear (unpigmented) or black (pigmented with carbon black). The former type of film permits sunlight to raise the soil temperature a few degrees. The Latter is an effective mulch (preventing growth of weeds) and is also much longer lasting, since the carbon black screens out the ultraviolet light which otherwise causes degradation of the polyethylene. My area of specialization is concerned with black plastics film and with carbon black itself.

As is common elsewhere, preparation of pigmented plastics in Mexico is divided into preparation of concentrates, or <u>masterbatches</u>; and preparation of the film. This enables the film manufacturer to avoid the problems of handling loose pigments. For agricultural applications, the masterbatch typically contains 25% carbon black, and this is let down to 2.5% in the film. It is essential for the masterbatch producer to achieve good dispersion of the carbon black in the masterbatch, since otherwise good dispersion cannot be achieved in the film. Poor dispersion in the masterbatch leads to serious problems in extrusion and blowing of the film, and to poor effectiveness and short lifetime (under exposure) of the film.

The problems of the masterbatch producer (relevant to the subject of this mission) may be classed as follows.

(1) Raw Materials. Problems arise from the limite number of grades available and from the uncertain level of quality control. Mexico has two carbon black producers, who are oriented toward the rubber industry, since (on a world-wide basis) rubber consumes 90-95% of carbon black production. Consequently, grades of carbon black which have been specially developed (in more developed countries) for plastics applications are not produced in Mexico, and would be difficult to import. (I do not regard this as a major problem, however, since it should be possible to use available grades with only a slight loss in efficiency for absorption of visible and UV light). Quality control of carbon black by the carbon black producer must also be oriented toward rubber applications; and aspects which may be especially important to the plastics manufacturer, such as pellet hardness and grit, may not be adequately controlled. I have no personal knowledge of the effectiveness of quality control on a day-by-day basis (according to the standard rubber-oriented tests) by the Mexican carbon black manufacturers. Regular testing at the carbon black plant gives the best assurance of high chality of production; and likewise, consumers of carbon black can best assure themselves of consistent quality by regular testing of their incoming shipments.

(2) Mixing Equipment and Method of Use. Carbon black is of finer particle size than all other pigments and consequently ismore difficult to disperse. Masterbatch producers who are set up for handling a variety of pigments may not have the high-shear mixers which are preferred for carbon black, and may not be familiar with the modifications and method of operation required to achieve optimum results with **car**bon black on the equipment which they do have.

(3) Assessment of Masterbatch Quality. As with any manufactured product, the masterbatch must be tested regularly as part of a quality control program. It is especially important to test for loading (concentration of carbon black), dispersion quality, ease of let-down with polyethylene, and optical properties (hiding power). Suitable methods must be set up and implemented.

Problems of a masterbatch producer were illustrated to me in connection with a visit to a particular producer.

The technical problems of a film producer include specification and testing of the raw materials (in his case, the polyethylene resin and the masterbatch, for which he may specify the grades of carbon black and polyethylene), processing (mixing, extrusion, blowing), and testing of the finished product. In the time available, and in view of my field of expertise (carbon black), it seemed best for me to concentrate on the problems of the masterbatch producer rather than those of the film producer. However, I was able to offer some suggestions to CIQA regardi - hoice of carbon black etc.

I spent most of my work period at the Centro de Investigacion en Quimica Aplicada (CIQA), in Saltillo, Coahuila. This organization has been functioning as a national technical center for plastics, including agricultural applications. In most respects CIQA is well set up for this role. The staff members with whom I had contact are bright, eager, and knowleigable in polymers including plastics. CIQA is generally well equipped for polymer studies, with good equipment for mixing, processing, physical testing, and fundamental analysis of polymers. Of course, new equipment is always desirable, and in connection with physical testing I made specific recommendations (based on personal experience) for dynamic mechanical testing equipment.

CIQA has an excellent policy of active cooperation with industry, universities, and consultants. Thus, CIQA personnel are cognizant of the problems of Mexican industrial companies and are not simply working in an ivory tower. CIQA brings its consultants (including myself) to visit companies with which it has a working relationship, thus enhancing that relationship and giving the consultants direct insight into the companies' problems.

In the particular field of plastics in agriculture, CIQA is in an especially favorable position due to the fact that it includes departments of plant biology.

A principal deficiency of CIQA is in the field of colloid and surface science, where CIQA has neither the specialized personnel nor the equipment. (My own visit, of course, was requested by CIQA in response to this perceived need). Another deficiency, perhaps inevitable in a new (8 years old) organization is the lack of older people with practical experience in industry. Some specific recommendations to correct these deficiencies are given at the end of this report.

ACCOMPLISHMENTS OF THIS MISSICN

Prior to my visit CIQA had obtained (from film producers) a few samples of "good" and "bad" commercially produced masterbatch. However, the reason for the difference in quality was not known. The test used by CIQA for evaluating the masterbatches consisted of letting down with polymer in an extruder-mixer and blowing a film for optical inspection. This test, while widely used, does not differentiate in a clear-cut manner between poor dispersion of the carbon black in the masterbatch and poor let-down of the masterbatch. CIQA had also carried out an experimental study of a number of variables in the preparation of masterbatch and of blown film. Mc t of this study appeared very worthwhile, but there was an anomaly in the behavior of two of the grades of carbon black (the coarser black was found to disperse less well).

The accomplishments of this mission may be grouped under the headings of the Post Description and some additional headings.

(1) Techniques Used for Dispersion of Inorganic Fillers in Plastics.

(a) I lectured for 4 hours to members of the CIQA staff and also to several students from the Universitad Autonoma de Coahuila, who had been invited by Dr. Ramos of CIQA. My lectures included a description of the nature of carbon black, grades and test methods, and the principles and techniques of dispersing carbon black in polymers.

(b) I advised Dr. Ramirez (of CIQA) on his experimental study (v. s.). In particular, it appeared likely that the anomaly in the behavior of the two grades of carbon black rould be due to the fact that they were obtained from two different producers.

(2) Methods for Evaluation of the Dispersion of Inorganic Fillers in Plastics.

(a) My lectures included a survey of the methods used for evaluation of the dispersion of carbon black in plastics. Methods used for evaluating dispersion in rubber were also discussed and the problems associated with application of these methods to plastics were brought out. Examples were also given of measurement of dispersion of other fillers. (b) I introduced the tape extrusion method for measurement of dispersion in the masterbatch. This is the preferred methor, widely used in industry for this purpose. Generally the masterbatch is extruded through a special tape die, at a thickness of 0.1 - 0.5 mm. CIQA did not have a tape die; but I found that the film blowing extruder, used at CIQA for extruding and blowing film from pure or lightly loaded polyethylene, could also be used with masterbatch. It was necessary to dry the masterbatch (e. g. 2 hours at 90° C) before extrusion, to avoid bubbles. The technique worked very well on the limited number of samples available.

(c) I worked with CIQA personnel on the methods for study of dispersion in agricultural plastics film; in particular, on the "squaeze-out" technique for microscopy, and on the interpretation of the appearance of various types of poor dispersion.

(d) Since some of the Mexican masterbatch producers may not be set up to carry out the tape extrusion test, it seemed desirable to develop a method which could be carried out with simple, inexpensive (or generally available) apparatus. One approach tried was to press a sheet from the masterbatch and examine the surface of a cut or fractured edge. Morking with CIQA mersonnel we were able to press good sheets, but various methods of cutting and fracture failed to give consistent differentiation betweem masterbatches of different quality. Another approach, based on the work of Dr. Ramirez of CIQA, was then investigated. This was to extrude the masterbatch through a "melt index" apparatus, which is widely used in the plastics industry for evaluation of resin viscosity, and then measure the elongation-to-break of the extrudate. We confirmed the possibility of extruding masterbatch through the melt index apparatus, but my time at CIQA expired before we were able to pursue development of the test. I arranged with Dr. Ramirez that he would continue with this work and that I would be available for consultation on it by mail.

(e) I brought with me a number of reprints of published articles and technical bulletins on measurement of dispersion, some of which I left at CIQA, while others were Xeroxed at CIQA.

(3) Evaluation of Plastics Film for Agricultural Use.

CIQA has an on-going program on this topic and there did not seem to be much that I could contribute to it. I did bring some pertinent reprints and I did make the recommendation that with the best grade of carbon black available in Mexico, the concentration of carbon black should be increased slightly over that normally used with the somewhat better grade available elsewhere (e. g., from 2.5% to 3.0%).

(4) Visit to a Masterbatch Producer.

CIQA arranged for Dr. Ramirez and me to visit Recubrimientos Plásticos, in Xalostoc, Edo. de México. This is one of the smaller producers of plastics masterbatch, but with an enterprising director, Ing. F. Gutiérrez C., with whom CIQA has established a good working relationship. This visit, which took place on November 29 and 30, gave me some insight into the problems faced by such producers. I made some specific recommendations

4

to Mr. Gutierrez regarding evaluation of raw materials and masterbatches, also with regard to a problem of segregation of carbon black and resin in feeding the continuous mixer. We also had a good discussion about carbon black, especially the differences between grades, the possible sources of problems in dispersion, and the optical properties. I also made a few suggestions concerning problems with non-black pigments.

(4) Quality Control of Raw Materials.

It became apparent to me that some of the problems of the masterbatch producer may stem from inadequate or non-existent quality control of raw materials. especially the carbon black. The best approach for me to try to rectify this situation seemed to be to advise CIOA in setting up test methods with which CIQA could become familiar, so that CIQA could then apply these methods to testing of suspect lots of carbon black from masterbatch producers and eventually encourage these producers to adopt some of these tests themselves. For this purpose it seemed best to use ASTM tests, since these are internationally recognized and should be acceptable to both manufacturers and consumers of carbon black. I advised CIQA regarding ASTM tests D1510, D1513, D1514, D2414, D3265, and D3313. I also promised to send information regarding a non-ASTI test for "structure" of carbon black (by specific volume under compression) which I have worked on in the past. I believe that these tests should enable CIQA to check on whether particular lots of carbon black are within specifications, and also to learn whether certain properties, such as pellet hardness, which may be acceptable for rubber customers may cause problems for plastics manufacturers. I agreed to continue to advise CIQA by mail regarding these tests and other aspects of this mission.

(5) Miscellaneous.

(a) I advised Dr. Ramos and Dr. Angulo concerning a proposed paper of theirs on bound rubber (formed by interaction of various elastomers with carbon black during mixing).

(b) I advised Dr. Ramirez concerning his proposed prover on the effect of variables in the production of black masterbatch and blown film.

(c) I agreed to assist Dr. Ramos in his search for candidates for a temporary position in polymer chemistry/engineering for which he has obtained funding.

RECOMMENDATIONS FOR FURTHER ACTION

(1) Technical.

(a) CIQA should gain further experience with the tape extrusion test for black masterbatch dispersion quality, using internally generated samples.

(b) CIQA should continue with a limited effort to develop a test for

quality based on simpler or more readily available equipment.

(c) CIQA should set up tests for monitoring carbon black quality. The cooperation of carbon black manufacturers should be sought in connection with establishment of these tests.

(2) Programs.

(a) CIQA should continue with its policy of cooperation with producers of masterbatch and film.

(b) CIQA should continue with its program of evaluation of plastics film for agricultural applications.

(c) CIQA should set up a program of regular monitoring of carbon black samples obtained from (i) carbon black manufacturers and (ii) carbon black consumers such as masterbatch producers.

(d) CIQA should be in a position to evaluate masterbatch quality for both producers and consumers of masterbatch, and also to evaluate film quality for producers and consumers of film. The extent to which CIQA should act as an arbiter of quality in case of dispute would depend on a number of political and legal aspects with which I am not familiar.

(3) Equipment.

CIQA should purchase additional equipment along the following lines (listed below in order of decreasing priority from the standpoint of this mission).

(a) Equipment necessary for implementation of the ASTM tests for carbon black (v. s.).

(b) Better microscope for visible light.

(c) Equipment for dynamic mechanical testing of polymers.

(d) Electron microscopes ("EM, SEM).

(4) Organizational.

(a) CIQA should hire a colloid/surface scientist as a full-time staff member.

(b) CIQA should try to hire retired technical people with practical experience, such as in technical service or production, on a part-time basis. People from more technically advanced countries could be very useful as consultants to CIQA and to Mexican industry; and by using them on a part-time basis, a greater diversity of backgrounds could be obtained for the same expenditure.

> Respectfully submitted. aron Medalo Avrom I. Medalia

December 24, 1984