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Standard Test Methods for Carbon Black—Dispersion in Rubber[1]

This standard is issued under the fixed designation D2663; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover the degree of dispersion of carbon black in rubber. Four test methods are described as follows:

	Sections
Test Method A—Visual Inspection	3 – 11
Test Method B—Agglomerate Count	12 – 22
Test Method C—Microroughness Measurement with Profilometer	23 – 33
Test Method D—Microroughness Measurement with IFM	34 – 42

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

2.2 *ASTM Adjuncts:*

Carbon Black Dispersion Standards[3]

Carbon Black Dispersion Chart[4]

TEST METHOD A—VISUAL INSPECTION

3. Scope

3.1 Test Method A is a qualitative visual test method. Ratings are made against a set of standard photographs (Fig. 1),[3] and the results are expressed on a numerical scale. This test method cannot be used for compounds that contain fillers other than carbon black.

4. Summary of Test Method

4.1 The compound rubber is torn or cut to expose a fresh surface for examination by the eye, aided preferably by a hand lens or a low-power binocular microscope. The dispersion level of the carbon black is compared against a series of five photographic standards and then rated numerically from 1 (very low) to 5 (high) (see Fig. 1).

5. Significance and Use

5.1 Visual dispersion ratings correlate with certain important physical properties of the compound. A rating of 5 indicates a state of dispersion developing near maximum properties, while a rating of 1 would indicate a state of dispersion developing considerably depressed properties. Normally, the visual dispersion ratings indicate the following levels of compound quality:

Visual Dispersion Rating	Classification
4 to 5	High
3 to 4	Intermediate
2 to 3	Low
1 to 2	Very low

6. Apparatus

6.1 *Sharp Knife or Razor Blade.*

6.2 *Hand Lens* (10×) or binocular microscope (10 to 20×).

6.3 *Illuminator,* microscopical-type.

[1] These test methods are under the jurisdiction of ASTM Committee D24 on Carbon Black and are the direct responsibility of Subcommittee D24.71 on Carbon Black Testing in Rubber.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from ASTM International Headquarters. Order Adjunct No. ADJD266302. Original adjunct produced in 1967.

[4] Available from ASTM International Headquarters. Order Adjunct No. ADJD266301. Original adjunct produced in 1967.

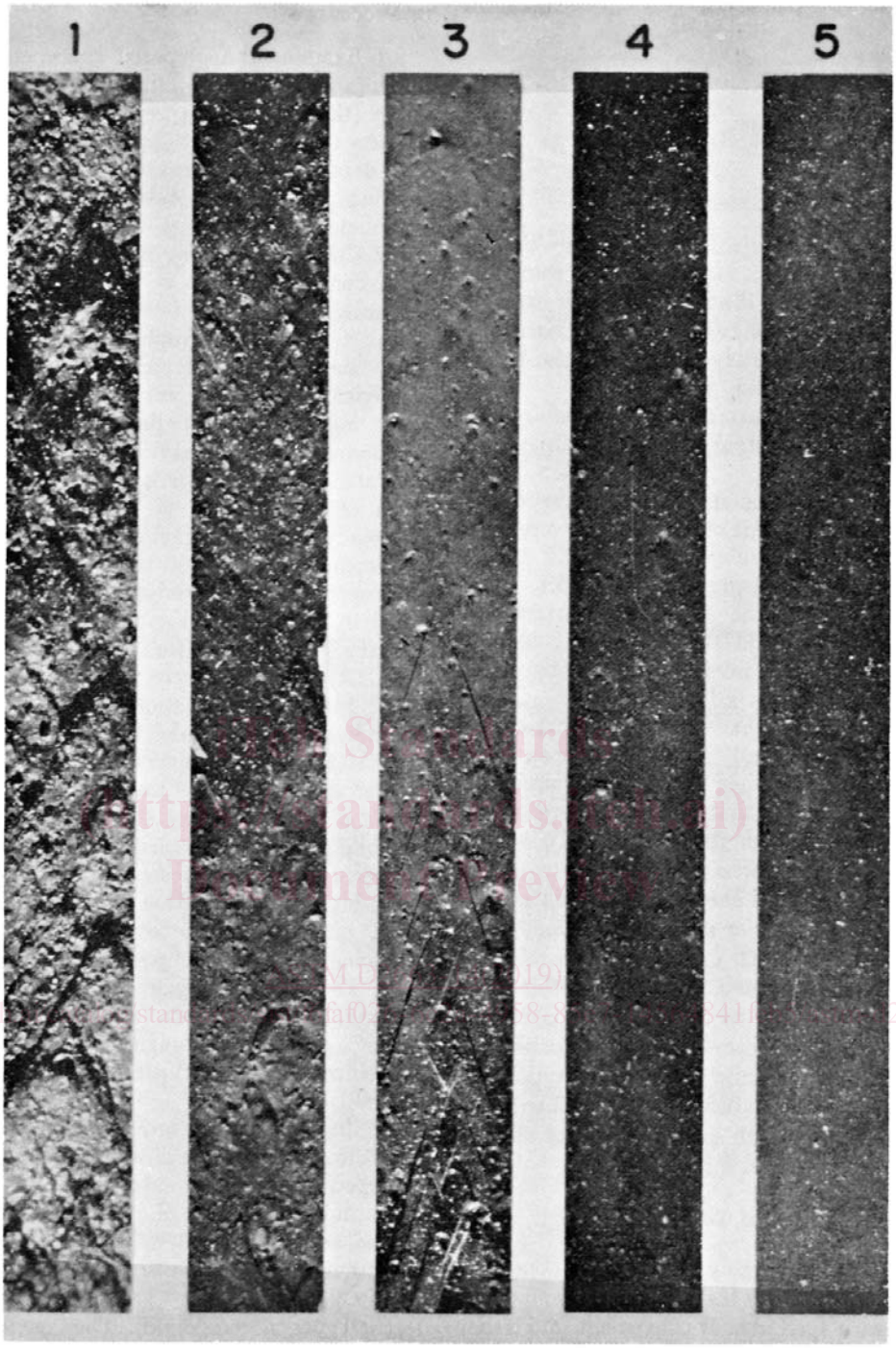

FIG. 1 Carbon Black Dispersion Standards—Visual Analysis of Torn Vulcanizates

6.4 *Knife Heater.*

6.5 *Series of Photographic Standards,* rating 1 to 5. These standards give the following percent dispersion ratings by the Agglomerate Count Method:

Visual Rating	Black Dispersed, %
1	70
2	80
3	91
4	96
5	99

7. Test Specimen

7.1 *Vulcanized Compounds*—Use a slab of rubber about 2 mm in thickness. Tear it so that a fresh surface is exposed. The tear may be initiated by a small cut. The most nearly flat part of the tear is used for rating.

7.2 *Unvulcanized Compounds*—Unvulcanized rubber may be examined as follows:

7.2.1 If the specimen contains curing agents, sheet it out and cure in a press to form a vulcanized slab about 2 mm in

thickness. Mill and cure in accordance with Practice D3182. Then proceed as in 7.1.

7.2.2 If the specimen contains no curatives, add the appropriate materials with a minimum of mixing. Then cure and proceed as above.

7.2.3 If the specimen contains no curatives and a dispersion evaluation with no further mixing is required, the compound must first be compressed to remove most of the air holes. To accomplish this, press the rubber into a slab between thin sheets of plastic in a mold at a pressure of about 1.03 kPa for 5 min at 105°C. Care should be taken to avoid excessive flow during this step. The surface to be examined is formed with a smooth cutting stroke using a sharp, hot knife (a standard type knife heater may be employed). The most nearly smooth and flat part of the cut surface is used for rating.

8. Number of Tests

8.1 Preferably more than one test (on different tears) should be made for each specimen. If convenient, more than one operator should rate the samples.

9. Procedure

9.1 Examine the prepared specimens under a hand lens or binocular microscope (the latter being preferred), with oblique illumination to accentuate surface detail. Keep the magnification and lighting conditions constant for all specimens.

9.2 Compare the size and frequency of carbon agglomerates in the specimens (showing up as surface bumps or depressions) to the photographic standards. Then assign the most closely matched numerical rating to each compound being rated. In borderline cases, use fractional ratings, for example, 3½ would indicate a rating between 3 and 4. In cases of dissimilarity in the size and frequency of the agglomerates in the specimen and those of the standards, the operator shall assign the rating that in his judgment is most applicable. Certain compounds (for example, NR and IR) are particularly prone to very small black agglomerations which are difficult to resolve by the Visual Inspection Method. In instances of high agglomerate frequency, the surface of stocks of this type may show a general roughness or fine pebbled appearance. Differences are best resolved at somewhat higher magnification (for example, 20×, binocular microscope). If at all possible, examine compounds of this type also by the agglomerate count method, at least until sufficient experience is gained to recognize dispersion differences with the Visual Inspection Method.

9.3 In comparing a series of different compounds, it is also desirable to rate the specimens side by side rather than one at a time. This use of a control compound is also advisable. This is best prepared by individual operators, since dispersion requirements may vary greatly for different types of compounds. The control sample should represent a minimum acceptable dispersion level for the type of compound being rated. Because it can be observed side by side with unknown samples under identical conditions, a control compound is more accurate than the photographic standards in discerning small deviations from what is considered the norm for a specific type of compound. Prepare a fresh surface on the control as often as necessary to ensure cleanliness.

10. Report

10.1 *Ratings:*

10.1.1 List all ratings, including those on any control compound, on the basis of the 1 to 5 scale defined by the standard photographs. Use fractional ratings when necessary.

10.1.2 Average the ratings on different specimens of the same compound as well as the ratings of different operators. Report the final average values.

10.2 *Compound Identification:*

10.2.1 *Formulation*—Whenever possible list the following:

10.2.1.1 Carbon black, type and loading,

10.2.1.2 Other fillers, type and loading,

10.2.1.3 Polymer type, and

10.2.1.4 Extender oil, type and loading.

10.2.2 *Mixing*—Describe the mixing of the compound in terms of one or more of the following:

10.2.2.1 Standard mixing procedure,

10.2.2.2 Type of equipment,

10.2.2.3 Masterbatch,

10.2.2.4 Finished compound (vulcanized), and

10.2.2.5 Finished compound (unvulcanized).

11. Precision and Bias

11.1 No statement is made about either the precision or the bias of Test Method A since the result is qualitative and not applicable to statistical treatment.

TEST METHOD B—AGGLOMERATE COUNT

12. Scope

12.1 Test Method B is a quantitative test method. Dispersion is evaluated by measuring with a light microscope the percentage area covered by black agglomerates in microtomed sections of the compound. Since this test method involves direct measurement, it is quantitative and more accurate than the visual test method. The test is applicable to the analysis of carbon black dispersion in compounds that contain other fillers.

13. Summary of Test Method

13.1 The compounded rubber is microtomed into sections sufficiently thin to permit observation of the carbon agglomerates by transmitted light, with the aid of a light microscope. The total cross-sectional area of all agglomerates 5 µm or larger is counted, and from the known content of carbon black in the stock, the percentage of carbon black below the 5-µm size is calculated and expressed as "Percentage of Carbon Black Dispersed."

14. Significance and Use

14.1 Certain important physical properties of the compound are influenced significantly by the degree of carbon black dispersion within the compound (for example, tensile strength and abrasion resistance). The correlation of these properties with the percentage dispersion determined by the Agglomerate Count Method approximates the following pattern for many types of black loaded rubber compounds:

FIG. 2 Rotary Microtome with Cryogenic Attachment for Sectioning Rubber Specimens

Dispersion, %	Classification
Above 99	Very high
97 to 99	High
95 to 97	Intermediate
92 to 95	Low
Below 92	Very low

15. Apparatus

15.1 *Microtome*—A rotary microtome[5] capable of producing sections from samples up to 3 mm in cross-section and 1 cm in length. Tungsten carbide knives are recommended. (See Fig. 2.)

15.2 *Cryogenic Cooling Unit*—A cryogenic cooling attachment for the above rotary microtome[6] capable of cooling the sample to −160°C. (See Fig. 2.)

15.3 *Microscope*—An optical microscope with binocular viewing and digital image capture is recommended. This should include a movable specimen stage and white light source with variable intensity. Lenses should include two 10× wide field eyepieces and objectives in the range from 6 to 10×. Taking into account microscope tube corrections, objectives should be selected so that magnifications in the range from 75 to 100× are available. (See Fig. 3.)

15.4 *Computer*—A computer should be available and interfaced to the digital camera on the microscope to capture digital photomicrographs of the specimens. (See Fig. 3.)

15.5 *Image Analysis Software*—Suitable image analysis software to allow thresholding of the captured micrographs, conversion of the thresholded image to binary and area fraction determination from the binary images. Examples of this type of software include, but are not limited to, Image J, ImagePro, NIH Image, IDL, and NIST Lispix.

15.6 *Razor Blades.*

[5] Example, Leica RM2265.
[6] Example, Leica LN22.

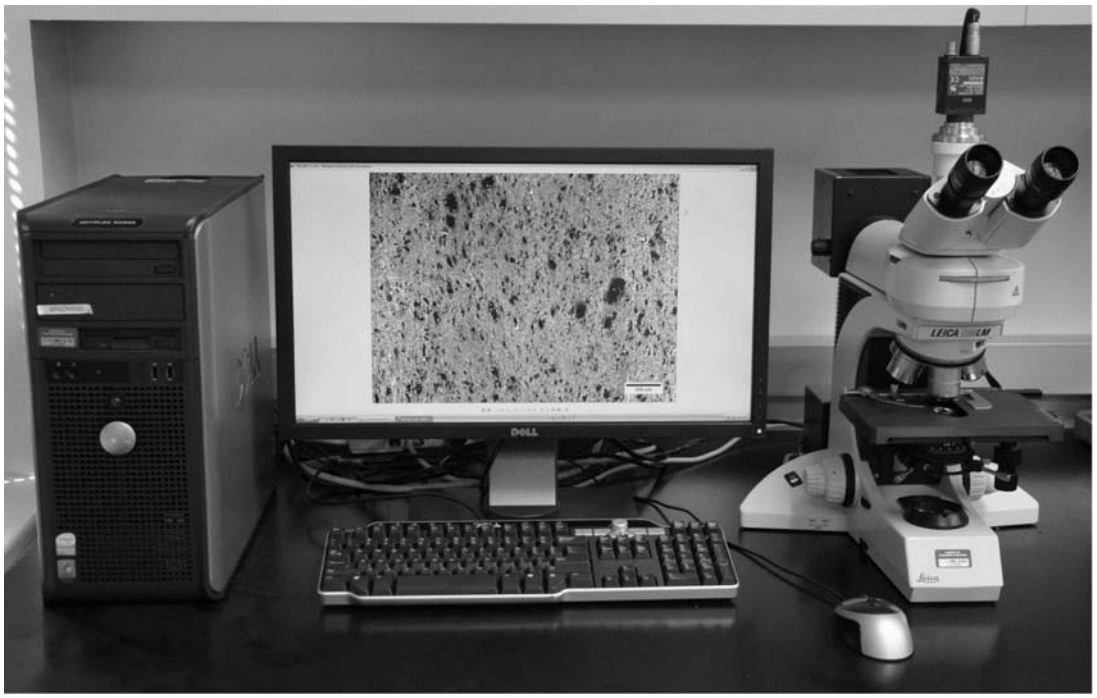

FIG. 3 Light Microscope Equipped with Digital Camera and Computer System

15.7 *Sable Brushes* (00).

15.8 *Microscope Slides and Cover Glasses.*

16. Reagents and Materials

16.1 *Liquid Nitrogen.*

16.2 *Organic Solvents*—Appropriate organic liquid to aid in flattening section onto the glass microscope slides. Examples include xylenes, toluene, and methanol.

17. Sampling

17.1 *Vulcanizates*—Specimens may be cut from standard test sheets (about 2-mm thick) or from pieces of actual cured articles. Vulcanized samples must be employed because of the solvent used to uncurl the thin sections. If pieces other than 2-mm sheets are used, they should first be cut down to a thickness of about 2 to 3 mm.

17.2 *Unvulcanized Compounds*—For rubbers of high unsaturation (for example, OE-SBR, NR, and BR), dust small bits (enough subsequently to form buttons about 10 mm in diameter and about 2 to 3-mm deep) thoroughly with dicumyl peroxide. Cure in a button mold[7] under high pressure at about 155°C. OE-SBR rubbers require about 30 to 60-min cure. BR requires about 10 to 15-min cure. After cure, scrape off the excess peroxide from the sample surface and proceed with sectioning in the standard manner, taking care not to pare down below the cured surface layer.

17.2.1 For IIR, satisfactory surface cures can be obtained with a mixture of 1 part tetramethylthiuram disulfide (TMTD), 1 part mercaptobenzothiazole (MBT), 1 part sulfur, and 5 parts zinc oxide, with a cure of 1 h at 155°C. Other alternative approaches for curing high unsaturation polymers without actually mixing in curatives are (*1*) high-energy radiation and (*2*) chemical treatment with sulfur monochloride. However, before using either of these latter methods, the stock should be pressed out to eliminate most of the air holes. Cure in accordance with Practice D3182.

18. Test Specimen

18.1 Cut out a specimen approximately 1 cm long, 1 cm wide, and approximately 2-mm deep.

18.2 Cut the square block into a trapezoidal shape that will fit the sample chuck on the rotary microtome.

18.3 Prepare one specimen block for each different compound to be examined.

19. Procedure

19.1 *Microtome Preparation*—Turn on the rotary microtome, insert the knife into the microtome and adjust to the correct cutting angle (see microtome manufacturer instructions). Fill the liquid nitrogen dewar and attach to the cryogenic chamber on the microtome. Cool the microtome chamber and knife holder.

19.2 *Sample Preparation*—Insert the prepared specimen block into the microtome chuck and insert the chuck into the microtome such that the long axis of the specimen is parallel to the cutting direction. Cool the sample to approximately 50°C below the elastomer glass transition temperature.

19.3 *Microtome Operation*—Manually advance the specimen so that the cutting face almost reaches the knife. At this point, with the advance set in increments of 5 to 10 μm, start

[7] A special mold containing several circular cavities that are approximately 10 mm in diameter and 3 mm deep.

microtoming until the specimen is faced level and full-size sections are being cut.

19.4 *Cutting Thin Sections*—After facing is complete, set the microtome control to the appropriate thickness depending on the carbon black loading. For standard elastomer compounds a thickness of 1 to 2 µm is a good starting point. Cut 4 to 5 sections, which will likely roll up, and allow the sections to collect on the back side of the knife and knife holder.

19.5 *Mounting Sections on Microscope Slides*—Using a clean, dry sable brush transfer a section from the knife block to a clean microscope slide placed on the edge of the microtome cryo-chamber. The section will be curled up in a small tight roll and should adhere to the brush with static electricity. Using a second sable brush, add a few drops of the organic liquid to the section. With careful manipulation of the solvent wet brush, unroll and spread the section out flat on the slide. An additional brush or small pointed stick may be helpful to roll out the section. Continue brushing gently to remove all wrinkles. Small amounts of additional solvent may be added as needed.

19.6 Repeat steps 19.4 and 19.5 until a sufficient number of sections have been brushed out. Then cover the sections with cover glasses or another glass microscope slide, and seal with tape, or a bit of cement at each corner.

19.7 *Preparing for Counting*—Inspect the sections for quality under the light microscope, and select one that is relatively free of wrinkles, holes, and knife marks. Also avoid sections that are very thin as some of the clumps of carbon black may be brushed out. If the sections are too thick or have too many wrinkles, holes or knife marks, adjust the microtome accordingly and produce additional sections.

19.8 Once good sections are obtained, remove the specimen from the microtome and measure the length and width of the faced block where the sections were obtained. The product of these dimensions is the area before swelling. Also, measure the length and width of a few of the sections mounted on the glass slides. Average these dimensions and their product is the section area after swelling. Record this value along with the sample area before swelling.

19.9 *Micrograph Acquisition*—Place the slides in the light microscope in transmission mode and select the magnification. Magnification should be in the range from 75 to 100× but the exact figure is left to the discretion of the individual operator, based on the specifications of his own particular microscope and lens system. Within the limits of 75 to 100×, the percent dispersion rating on a given section will not change significantly, provided that sampling is adequate. However, magnification should be kept constant in comparing and classifying agglomerate size within different samples. Adjust the lighting and exposure conditions to obtain good images and acquire ten non-overlapping images showing the carbon black agglomerates in the elastomer matrix (Fig. 4). Save the micrographs in a non-lossy (uncompressed image in order not to lose micrograph information) file format.

19.10 *Micrograph Analysis*—In an appropriate image analysis software package, open the first micrograph. To analyze the images, the first step is to threshold the image such that the carbon black aggregates are isolated from the background (usually brown in color). Care should be taken to minimize the number of defects (knife marks, folds, etc.) that are included in the area selected by the threshold operation. Once the threshold is complete, a binary image will be generated (Fig. 4). Using the appropriate software tool, the agglomerates greater than 5 µm in size should be counted and a total area fraction of these agglomerates calculated. Repeat this analysis for each image and average the ten area fraction values together to obtain the overall agglomerate area fraction.

20. Calculation and Interpretation of Results

20.1 *Percent Dispersion*—Calculate the percent dispersion, representing the percentage of carbon black that has been dispersed below the 5-µm agglomerate size, as follows:

$$\text{Dispersion, \%} = 100 - SU/L$$

where:

U = agglomerate area fraction. (This represents an average of the ten area fraction measurements on the sections. See Note 1.)

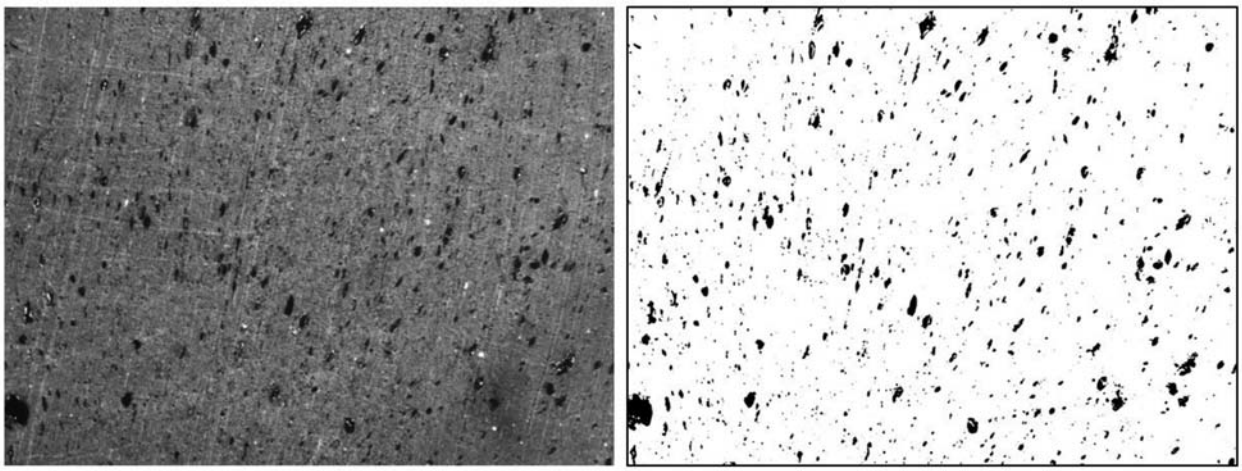

FIG. 4 Left: Light micrograph showing the carbon black agglomerates (dark regions) in a rubber sample. Right: The binary image produced from the micrograph after thresholding to isolate the carbon black agglomerates