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**RECLAIMED RUBBER**

BY

A. T. McPHERSON

CIRCULAR OF THE BUREAU OF STANDARDS, No. 393

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By A. T. McPherson

## ABSTRACT

Reclaimed rubber is a material, made from scrap or waste rubber, which is designed to replace or to supplement new rubber in the manufacture of rubber products. The larger part of the reclaimed rubber produced is made by the alkali process in which ground scrap rubber is digested with sodium hydroxide solution and softeners under pressure for the threefold purpose of destroying fabric, removing free sulphur, and plasticizing the rubber. The product is washed, dried, refined, strained, and sometimes compounded before marketing. Some reclaimed rubber is produced by the acid process, which differs from the alkali process chiefly in that the ground scrap is digested with sulphuric acid solution in order to destroy the fabric, and is subsequently treated with steam under pressure to plasticize the rubber. Reclaimed rubber contains most of the constituents of the scrap rubber and, in addition, softeners, fillers, and other substances which may be added during reclaiming. Reclaimed rubber is not equal to new rubber in strength, stretch, or resistance to abrasive wear. However, it may be used satisfactorily for many rubber products in which softness and flexibility are important. In manufacturing operations reclaimed rubber can be processed more readily and at less cost than crude rubber. The consumption of reclaimed rubber fluctuates with the price of crude rubber. Present reclaiming processes are not so well adapted to small-scale operation as are direct means of utilizing discarded rubber products.

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## I. INTRODUCTION

This circular has been prepared to meet requests for information about reclaimed rubber. The aim has been to give a description of processes for the reclaiming of rubber that are in current use in the United States, and to review some of the properties and applications of the material that may be of general interest. More attention has been paid to the principles of reclaiming operations than to details of machinery and equipment.

It has not been possible, within the scope of this circular, to explain all technical terms and references to processes of rubber manufacture or testing so as to be clearly understood by a person unfamiliar with rubber technology. For general information of this nature reference should be made to treatises on rubber which may be found in practically any large library. The testing of rubber and methods of manufacture are described briefly in Circular of the Bureau of Standards, No. 38, which may either be consulted in libraries or purchased from the Superintendent of Documents, Washington, D. C. (Price, 30 cents; stamps not accepted.)

The reader who seeks more detailed information about reclaiming processes and properties of reclaimed rubber is referred to the list of recent publications given in the last section of this circular. Statistics regarding the production, consumption, and price of reclaimed rubber may be obtained from the Bureau of Foreign and Domestic Commerce, Washington, D. C. Current statistics and market reports are published regularly in the following rubber-trade journals:

India Rubber World, published monthly by the Federated Business Publications (Inc.), 420 Lexington Avenue, New York, N. Y.

Rubber Age, published twice monthly by the Palmerton Publishing Co., 250 West Fifty-seventh Street, New York, N. Y.

A discussion of patents relating to reclaimed rubber is beyond the scope of this circular. References to patents may be found in several articles cited in the bibliography. In this regard particular attention is directed to *A Review of Reclaiming Processes*, by C. E. Bishop, *Rubber Age*, volume 21, pages 247-248; 1927. This publication describes briefly the patents on reclaiming issued in the United States up to 1927.

## II. DEFINITION OF RECLAIMED RUBBER, AND THE SCOPE OF RECLAIMING PROCESSES

Reclaimed rubber is a material made from scrap or waste rubber by a softening and plasticizing process; it is designed to replace or to supplement new rubber in the manufacture of rubber products. Reclaimed rubber is sometimes spoken of as devulcanized rubber but this is a misnomer, because reclaiming does not reverse the vulcanizing process or remove from combination any significant proportion of the sulphur that was used to effect vulcanization. Reclaimed rubber differs materially from new or natural gum rubber both in composition and in properties.

The chief results achieved by all reclaiming processes are the separation of metal parts and fabric from waste rubber goods and the conversion of the rubber itself into a homogenous, plastic product. Some processes also effect the removal of the so called free sulphur, which is a portion of the sulphur employed for vulcanization that did not combine with the rubber.

Reclaiming also involves the addition of substances to rubber. Practically all reclaimed rubber contains softeners and plasticizers added in the course of reclaiming operations. A fair proportion of the reclaimed rubber that is produced is compounded by the manufacturer with additional softeners, fillers, and other materials in order to give the product certain properties or to adapt it better to specific uses.

Ground scrap rubber, sometimes known as shoddy,<sup>1</sup> is used in the manufacture of some rubber products. The production and use of this material is not considered in the present discussion, since no plasticising process is involved in its manufacture.

## III. COLLECTION AND CLASSIFICATION OF SCRAP RUBBER

The reclaimed rubber used in manufacture has amounted, in recent years, to between 40 and 50 per cent of the quantity of new rubber consumed, so a considerable proportion of the rubber that goes into rubber products is ultimately salvaged and used again. This comparison does not indicate the exact proportion of rubber that is recovered from waste because reclaimed rubber contains fillers, softeners, and other ingredients. On the basis of rubber substance the consumption of reclaimed rubber is probably equivalent to 25 or 30 per cent of the consumption of new rubber.

Within the limits of economical freight transportation practically all discarded automobile inner tubes and most worn-out casings find their way to the reclaiming factories. The recovery of many other types of scrap is less complete. The rubber articles most in demand

<sup>1</sup> The term, "shoddy," is sometimes used as synonymous with reclaimed rubber. However, it is here employed in a limited sense to refer to ground waste rubber.

for reclaiming are those which were largely or wholly made from new rubber.

Collection of scrap rubber is usually made by the local junk dealer. When sufficient stocks have been accumulated the scrap is sorted, baled, and sold to the dealer or trader in scrap rubber.

TABLE 1.—*Rubber scrap*[Consumers' buying prices, carload lots, delivered at eastern mills high and low prices, 1930 <sup>1</sup>]

Class	Variety	Quantity	Prices	
			High	Low
Boots and shoes.....	Boots and shoes, black.....	100 pounds..	\$1.20	\$1.10
	Untrimmed arctics.....	do.....	.75	.70
	Tennis shoes and soles.....	do.....	.75	.60
Inner tubes.....	No. 1, floating.....	Pound.....	.07 $\frac{1}{4}$	.04 $\frac{3}{4}$
	No. 2, compound.....	do.....	.03 $\frac{3}{8}$	.02
	Red.....	do.....	.04 $\frac{1}{8}$	.02
	Mixed tubes.....	do.....	.03 $\frac{3}{8}$	.01 $\frac{1}{2}$
Pneumatic tires.....	Mixed auto tires with beads.....	Ton.....	18.00	11.00
	Beadless.....	do.....	27.50	15.00
	Auto tire carcass.....	do.....	25.00	17.00
	Black auto peelings.....	do.....	30.00	20.00
Solid tires.....	Clean mixed truck.....	do.....	26.00	22.00
	Light gravity.....	do.....	31.00	27.00
Mechanicals.....	Hose, air brake.....	do.....	21.00	11.00
	Hose, garden, rubber covered.....	do.....	.00 $\frac{5}{8}$	.00 $\frac{3}{4}$
	No. 1, red.....	do.....	.02	.01 $\frac{1}{2}$
	No. 2, red.....	do.....	.01	.01
	White druggists sundries.....	do.....	.02	.01 $\frac{1}{2}$
Hard rubber.....	No. 1, hard rubber.....	do.....	.10 $\frac{1}{2}$	.08

<sup>1</sup> From market reports in India Rubber World.

The trader is the responsible agency to whom the reclaimer looks for a supply of properly graded and classified scrap, delivered in carload lots at the reclaiming plant.

The standard commercial grades and classes of scrap rubber used for reclaiming are shown by the market listing given in Table 1. Tires and inner tubes are the most important classes, and make up the bulk of the tonnage. Mechanical goods, boots and shoes, and hard rubber constitute other classes. New scrap rubber consisting of trimmings and waste from manufacturing processes is also reclaimed, but does not appear in the market quotations, since many manufacturers have their own reclaiming facilities while others make regular contracts with reclaimers for salvaging the rubber from their waste.

The highest and the lowest prices for the different classes and grades of scrap rubber which obtained during 1930 are given in the table to indicate the relative value of the various kinds of scrap. Except for hard rubber, the most valuable scrap is inner tubes of low specific gravity. The least valuable scrap is mechanical goods. This is due to the fact that most articles in this class were originally made from reclaimed rubber, which on a second reclaiming gives an inferior product. Since the prices quoted are for scrap rubber delivered at the mills it is evident that the margin of profit in the collection and handling must be small. As a matter of fact, it is profitable to gather scrap rubber only within a certain radius of a reclaiming plant, this radius being determined by freight rates and by prevailing market prices for scrap rubber.



## IV. DESCRIPTION OF RECLAIMING PROCESSES

### 1. CLASSIFICATION

Reclaiming processes are generally designated by the means which are employed to remove cotton fabric, since a large proportion of scrap rubber contains fabric. The main reclaiming processes on this basis are: (1) The alkali process, (2) the acid process, (3) the mechanical process, (4) solution processes, and (5) the viscose process. The larger part of the reclaimed rubber that is made from scrap which contains fabric is produced by the alkali process. The remainder is produced by the acid process. The other processes have been the subject of considerable experimentation, but so far as is known, none of them is operated for the reclaiming of vulcanized rubber.

The reclaiming of fabric-free scrap rubber is sometimes effected by the regular alkali process. In other cases, modifications of this process or simpler processes are used.

The alkali process will be described in some detail. The other processes will be described more briefly and reference will be made to the alkali process for those steps and procedures which they may have in common with it. Only passing reference will be made to the reclaiming of hard rubber.

### 2. THE ALKALI PROCESS

#### (a) PREPARATION OF SCRAP RUBBER FOR RECLAIMING

Preparatory to reclaiming, the metal parts must be removed from the waste rubber articles, and the rubber and fabric must be reduced to a state of subdivision suitable for the action of reagents. In the case of pneumatic tires the bead is usually removed by a machine specially designed for that purpose. Solid tires are either cut or stripped from their rims.

Various types of machines are employed for the chopping and grinding<sup>2</sup> of scrap rubber. Chopping machines make use of rotating knives which operate in a manner not unlike a lawn mower. These cut the debeaded tires and other scrap rubber into pieces from a few inches to a foot in length. The chopped rubber is usually ground by passing it between heavy rolls, which may be smooth or may have corrugated surfaces. A continuous sizing device screens the ground scrap rubber to the desired size and returns any large pieces to the rolls. In some plants the grinding is done by a fine grinding "hog" or a shredder, while in others both the coarse and the fine stages of the reduction are accomplished with "hogs." The scrap rubber is usually ground to pass through a screen having a  $\frac{1}{4}$ -inch mesh, though sizes as fine as  $\frac{3}{16}$  inch and as coarse as 1 inch may be employed.

The ground scrap rubber is usually passed over a magnetic separator to remove any small pieces of iron or steel that may be present, such as tacks or nails that may have been embedded in the tires from which the scrap was made.

#### (b) ALKALI DIGESTION

The most important step in the alkali process is the digestion of the ground scrap with a solution of caustic soda or sodium hydroxide at an elevated temperature under pressure. This accomplishes in

<sup>2</sup> A special type of scrap-rubber grinder is described in *India Rubber J.*, 74, pp. 297-298; Aug. 20, 1927.

one operation the destruction of the fabric, the plasticizing of the rubber, and the removal of uncombined or free sulphur. This process is frequently spoken of as "devulcanization."

The ground scrap rubber is charged into digestors or autoclaves with 4 to 6 per cent sodium hydroxide solution and softening agents for rubber. The digestors are heavy cylindrical vessels of 3,000 to 5,000 gallons capacity, provided with jackets for heating by means of steam under pressure, and fitted with agitators to keep the charge thoroughly mixed. In 1926 the caustic soda used amounted to about 13 pounds per 100 pounds of ground scrap, but a smaller proportion of caustic soda is said to be employed at the present time.

The softeners which may be employed are numerous and include light oils, heavy oils, tars, and pitches derived from the distillation of wood, petroleum, and coal, and also fatty acids, though the latter are more commonly added at a subsequent stage. The particular function of the softeners added during alkali digestion is to swell the rubber and to facilitate its conversion to a plastic material. The volatile softeners may, in large part, pass off with the wash liquor at the close of the digestion, whereas the nonvolatile softeners may remain in the rubber either in the form in which they were added or as sodium salts produced by the action of the caustic soda. No information seems to be available as to the amounts of softeners used; no doubt the proportions may vary within wide limits.

The temperatures employed for the alkali digestion ordinarily range from 178° to 198° C., being equivalent to steam pressures of 125 to 200 pounds per square inch. The time of digestion at the highest temperature may be as short as 8 hours; at the lowest temperature 20 hours may be required, or even longer if no softener is used.

#### (c) WASHING, DEWATERING, AND DRYING

After the digestion with caustic soda the ground scrap rubber is washed with water to remove the residual alkali as well as salts produced by the reaction of the alkali with sulphur and the disintegration products of the cotton fabric. As will be discussed subsequently, it is not practicable to wash all the alkali from rubber. In the course of the washing process the rubber is usually passed over riffles to remove sand, grit, and metal particles derived from the original scrap rubber. A considerable quantity of rubber is carried away in the wash water as fine particles. This rubber is recovered by allowing the wash water to settle in large tanks. The sludge is collected and most of the water is drained out of it by continuous vacuum-filtering devices or other means, after which it is returned to the main quantity of washed scrap.

The washed rubber is dewatered by means of screw presses or other devices which squeeze out as much of the water as possible by the application of pressure. The product is then dried in a current of heated air. In modern plants the moist rubber is spread in a thin layer on a wide screen which moves slowly through a drying chamber in a direction opposite to the current of warm air.

#### (d) MILLING AND COMPOUNDING

The dried rubber which comes from the screens as a sheet of loosely coherent particles is worked on roll mills or in an internal mixer to convert it into a homogeneous mass of uniform consistency.

This step in the process is called batching out. At this stage softening agents are sometimes added, as well as fillers. Both of these assist in working the rubber into a smooth, homogeneous batch, and as ingredients of the finished reclaimed rubber, they serve other purposes as well. The softeners added here are usually tars, pitches, and asphalts containing no volatile components. Stearic acid and other fatty acids may be added, not so much as softeners, as for their effect on the vulcanizing properties of the reclaimed rubber.

The fillers which may be employed include carbon black, small proportions of zinc oxide, and such materials as clay and whiting, which are cheap and have a relatively low specific gravity. The carbon black, by reason of its stiffening effect on the stock may assist materially in breaking up and working out lumps, thus improving the uniformity of the reclaimed rubber. It also increases the strength of the product. The zinc oxide, if used, is employed in small amounts for its effect on the vulcanization characteristics of the reclaimed rubber. The bulk fillers are employed to adjust the specific gravity to a desired figure and to increase the yield.

In some instances sulphur, accelerators, and antioxidants may be compounded with reclaimed rubber during manufacture, though these are less commonly added than fillers and softeners.

#### (e) REFINING AND STRAINING

The finishing steps of the reclaiming process, refining and straining, are designed to improve further the consistency and the homogeneity of the reclaimed rubber. Refining is accomplished by passing the rubber between heated rolls which may be set as close together as 0.003 inch. This either removes or breaks up any lumps of tough rubber which thus far may have escaped being plasticized and mixed. After passing through the refining rolls the product is usually strained by forcing it through a screen having 40 to 60 meshes per inch. The machine used for straining rubber is similar to a sausage mill, except that the screen is in the position occupied by the cutting knives of the latter. The straining takes out metal particles and other foreign objects which may have escaped removal previously; it also accomplishes still further mixing and plasticizing of the rubber. After straining, the product is again passed through refining rolls, and the thin sheet thus obtained is wound continuously around a drum, in a process known as leaf sheeting, until a layer one-half inch or more in thickness is built up. This layer is then cut off and opened flat to form a slab, which is dusted with talc to keep it from sticking, and packaged or baled for the market.

### 3. THE ACID PROCESS

In the reclaiming of rubber by the acid process the removal of fabric is accomplished by destroying it with hot sulphuric acid. The ground scrap rubber is mixed with 20 per cent sulphuric acid in lead-lined vats, and heated with steam at atmospheric pressure for four to six hours. The digested scrap is then washed with water to remove the disintegrated fabric and most of the acid, and passed over riffles to take out sand and grit. The product is then conveyed into a tank containing one-fourth of 1 per cent solution of caustic soda to neutralize the residual acid, is washed again and is dewatered by means of

centrifugal machines. The next step is a steam digestion for the purpose of softening and plasticizing the rubber. This operation is commonly spoken of as "devulcanization," though the designation is incorrect in the literal sense of the word. For the steam digestion the partially dried rubber is mixed with softeners and placed in steel boxes or pans which are stacked on small cars that are run into heavy-pressure vessels called "devulcanizers." Steam is admitted to the pressure vessels at 55 pounds per square inch or higher for a period which is usually 8 to 12 hours.

The rubber which has been softened by this treatment is dried, milled, compounded, strained, and refined by processes similar to those described in connection with the alkali process.

#### 4. THE MECHANICAL PROCESS

The mechanical process for the reclaiming of rubber is designed to separate fabric from ground rubber by some mechanical means, such as an air current. The situation in regard to this process is summarized by Winkelmann as follows:

Mechanical methods of separation of fiber from ground rubber have been tried from the beginning of the industry. About 1870-71 E. H. Clapp removed fiber by means of an air blast. Methods for separation of the fiber in satisfactory form and finding uses for it have always interested the manufacturer of reclaimed rubber. The grade of cotton used in rubber products is such that many uses can be found for it if the rubber content can be economically decreased to a very minimum. The separated fiber has always carried with it rubber particles of varying size, therefore limiting its applications. There is no question but what a machine will be developed at some future date which will remove practically all the rubber and make it possible to realize on the value of the fiber in the scrap.<sup>3</sup>

#### 5. SOLUTION PROCESSES

Various solution processes have been designed for the separation of rubber from fabric by means of solvents for the rubber. In some instances the processes are intended to separate fillers as well as fiber from the rubber. In general, the ground scrap rubber is treated with a solvent at an elevated temperature, and the resulting solution of rubber is separated from fabric, metal parts, grit, and the like by decantation or by filtration. The rubber may be recovered from the solution by evaporation or by precipitation.

The difficulty with this process lies in the lack of a suitable solvent. No solvent is known which will dissolve vulcanized rubber without at the same time radically changing its properties. The solvents which have been proposed include kerosene and other petroleum fractions, cymene, turpentine, toluene, xylene, pine oil, and many other organic liquids. When vulcanized rubber is heated with practically any of these liquids for a sufficient length of time at about 150° C. a fairly mobile solution is produced which may be separated from fiber with reasonable facility. But the rubber, when recovered from this solution, is so soft and tacky that it is not suitable for ordinary manufacturing purposes.

Solutions made from vulcanized rubber are very different in properties from the solutions of unvulcanized or natural gum rubber which constitute the familiar rubber cements. So far as is known, solutions of vulcanized rubber have no practical use.

<sup>3</sup> The History and Trends in the Use of Reclaimed and Scrap Rubber, *Rubber Age*, 25, p. 545; Aug. 25, 1929.

A solution process has recently been developed on a production basis for the recovery of both the cotton and the rubber from unvulcanized tire ply scrap. This scrap, which consists of trimmings from the manufacture of tires, is composed of uncured rubber and long staple cotton fabric. The rubber is extracted from the fabric by means of benzene, and the resulting solution is utilized in manufacture as a cement.

#### 6. THE VISCOSE PROCESS

The viscose process aims to salvage both the cotton fabric and the rubber of waste rubber articles by converting the former to viscose at the same time that the rubber is softened and plasticized. The viscose is not obtained as a separate product, but is produced in the rubber batch during reclaiming operations and becomes thoroughly dispersed in the rubber by the mixing, refining, and straining operations. The viscose is said not only to increase the yield, but also to improve the strength and durability of the reclaimed rubber.

The reactions involved in the conversion of cotton fabric to viscose are: (1) The production of a sodium derivative of cellulose by the action of sodium hydroxide or caustic soda on the cotton, (2) the formation of cellulose xanthate by the addition of carbon disulphide to the above, and (3) the regeneration of viscose by the action of acid or other reagents on the cellulose xanthate or by heating.

No published information on the viscose process seems to be available other than that relating to patents.

#### 7. PROCESSES FOR THE RECLAIMING OF FABRIC-FREE RUBBER

In the reclaiming of fabric-free rubber the above-described processes may be used with little or no alteration, though various simplifications may be effected by reason of the absence of fabric.

A very simple procedure is used in the reclaiming of rubber to be employed in making certain grades of hard-rubber goods. High-grade scrap rubber, such as inner tubes, is ground between heated rolls to produce a homogeneous, plastic mass; softeners are sometimes added in the course of this operation. The product may be incorporated directly into the batch composition for making hard rubber or ebonite.

Another process which may be applied not only to inner tubes but also to air bags and to solid tires is called the "direct steam heater and pan process." In this process the ground scrap rubber is usually mixed with softeners and placed in steel boxes or pans. These are covered and are stacked on cars which are run into a large pressure vessel known as a heater. The treatment consists in heating the charge in open steam under pressures from 75 to 125 pounds per square inch. The heating period is 5 to 20 hours, depending on the character of the material and on the steam pressure. The stock which has been softened by this treatment is milled, strained, and refined as previously described. In this process the free sulphur is not removed, but combines with the rubber under the influence of heat.

If the removal of free sulphur is desired, the regular alkali process may be used, much the same as for fabric containing rubber, or the "heater process with agitation" may be employed. In the latter

process the ground scrap is digested for several hours with a hot solution of sodium hydroxide at atmospheric pressure, this treatment being designed to remove the free sulphur. The product is then washed, partially dewatered, and charged into a pressure vessel which is heated indirectly by steam circulated in a jacket. The pressure vessel or heater is provided with an agitator so that the contents may be stirred to insure uniform heating. Though this heating is done in a closed container, part of the moisture present in the batch is permitted to escape as steam, so that at the end of the heating period the charge is sufficiently dry for milling and refining.

### 8. THE RECLAIMING OF SCRAP HARD RUBBER

There seems to be no recent published information on the reclaiming of hard-rubber scrap, but so far as is known the process consists essentially in separating the metal parts and other foreign materials and in converting the hard rubber to a clean, finely divided powder. Special care is taken to remove grit of any sort, since this would affect the finish of articles subsequently made from the powder or dust. Hard-rubber dust is extensively used in the manufacture of hard-rubber products; when employed for this purpose it may be incorporated in the batch composition along with rubber, sulphur, and other ingredients, or it may be used as a molding powder. By no means all of the hard-rubber dust that is required by the industry is produced from salvaged products; much of it—probably the larger part—is made from new hard rubber that is manufactured for the express purpose of being ground to dust.

## V. COMPOSITION OF RECLAIMED RUBBER

Reclaimed rubber contains virtually all the ingredients of the scrap rubber from which it was prepared except metal parts, fabric, and some of the sulphur. In addition, it contains a variety of materials added incidentally or intentionally during the course of reclaiming. The main components or classes of components will be considered in the following paragraphs.

### 1. THE HYDROCARBON OF RECLAIMED RUBBER

The basic constituent of reclaimed rubber is the plasticized product derived from the original vulcanized scrap rubber. This is a hydrocarbon combined with a certain proportion of sulphur. It should be emphasized that this material is not identical with, or closely similar either to vulcanized rubber before reclaiming or to crude or natural gum rubber. Although it has approximately the same ultimate composition as the former, the two materials differ both in chemical constitution and in properties. A larger proportion of reclaimed rubber is soluble in chloroform than of vulcanized rubber, and less bromine will combine with the former than with the latter, indicating that some of the double bonds in the rubber molecule disappear in the course of reclaiming.

Readers who are interested in the chemistry of reclaiming are referred for further information to a paper by H. A. Winkelmann entitled, "Present and Future of Reclaimed Rubber," *Industrial and Engineering Chemistry*, volume 18, pages 1163-1168; November, 1926.

## 2. SULPHUR

As was indicated in the discussion of reclaiming processes, the free or uncombined sulphur may be removed from scrap rubber by extraction with alkali, or it may be allowed to remain, in which case it combines with the rubber during the heating or digestion that is required to soften or plasticize the scrap. When sulphur is extracted from rubber by alkali the mechanism of the process involves the migration of the sulphur to the surface of the rubber, rather than the permeation of the rubber by alkali.

The sulphur that is combined with rubber is not affected appreciably by the long heating with alkali involved in reclaiming. Vulcanization involves the combination of sulphur with rubber, and if reclaiming were devulcanization in the exact sense of the word, the vulcanization reaction should be reversed and sulphur removed from combination. However, this is not accomplished. A small amount of the combined sulphur is removed, but this is due probably not so much to a reaction of the alkali as to thermal decomposition of the molecule of vulcanized rubber which, in the absence of caustic soda, would lead to the production of hydrogen sulphide.

## 3. FILLERS

Fillers present in scrap rubber go over into reclaimed rubber practically unchanged, even though they may be substances which are soluble in the acids or alkalies used for reclaiming. Zinc oxide, for instance, when alone is soluble in both sulphuric acid and sodium hydroxide, but when incorporated in rubber it is not attacked by either because the individual particles are surrounded by films of rubber, and rubber is not permeable to the reagents.

Study of the fillers in reclaimed rubber is not infrequently made for the purpose of ascertaining the type of scrap from which it was prepared. This is possible, since, for each class of rubber products, certain fillers are commonly used, and these are employed in a fairly definite range of percentages.

## 4. RESIDUAL FABRIC

The destruction of fabric by acid or alkali is dependent on the mechanical penetration of these reagents into the particles of ground scrap, starting at the edges where the fabric is exposed. Portions of the fabric which are well embedded in rubber may escape attack, or may be only partially destroyed, with the result that cellulose fibers or their degradation products may be found in the reclaimed rubber. In 1920 seven samples of reclaimed rubber were examined at the National Bureau of Standards,<sup>4</sup> and were found to contain from 0.8 to 11 per cent of cellulose. No recent data seem to be available relating to the amount of cellulose present in the reclaimed rubber that is produced at the present time.

## 5. RESIDUAL ALKALI

Sodium carbonate and other alkaline constituents which are left in rubber reclaimed by the alkali process have an important bearing on its use in manufacture. The amount of alkali normally present is

<sup>4</sup> S. W. Epstein, Determination of Cellulose in Rubber Goods, B. S. Tech. Paper No. 154.

only a fraction of 1 per cent, but it is significant because it is a powerful accelerator of vulcanization. Rubber stocks made with reclaimed rubber vulcanize more quickly than those made with new rubber. This in itself may not be objectionable, but variations in alkali content may cause trouble in manufacturing processes by leading to the production of articles which are either overvulcanized or undervulcanized and consequently do not possess the optimum mechanical or aging characteristics. The presence of relatively large proportions of alkali may produce such rapid acceleration as to cause scorching or partial vulcanization during mixing or processing operations.

Residual alkalies, together with other residual salts, have another disadvantage in that they increase the capacity of reclaimed rubber to absorb water, either on immersion or from the atmosphere. A high water absorption coefficient is objectionable in rubber products intended for electrical insulation.

#### 6. SOFTENERS

As was indicated in the description of reclaiming processes a wide variety of softeners may be introduced into reclaimed rubber. These may be present in the finished product either in the form in which they were introduced or as sodium salts, in case they were acidic in nature and were added prior to or during the alkali digestion. In addition to softeners proper, stearic and other fatty acids may be introduced into reclaimed rubber in amounts up to 1 or 2 per cent. It has been shown that fatty acids may decidedly improve the mechanical properties of products made from reclaimed rubber.

#### 7. ADDED FILLERS

While reclaimed rubber already contains the fillers that were present in the scrap, more fillers may be added. These include carbon black, whiting, clay, lithopone, zinc oxide, and various other common fillers. Several purposes may be served by the compounding of reclaimed rubber with fillers during manufacture. Reinforcing fillers, such as carbon black, may facilitate the working out of lumps and the production of a smooth, homogeneous, and consistent product. The bulk fillers, as whiting and clay, are frequently employed for the purpose of adjusting the specific gravity to a desired figure. However, the chief object in compounding reclaimed rubber with fillers is to adapt it better to specific uses. It was estimated that 20 to 30 per cent of the reclaimed rubber marketed in 1928 and 1929 had been compounded by the reclaimer. No information seems to be available as to the kind or percentage of fillers most commonly used.

#### 8. ANALYTICAL DETECTION AND ESTIMATION OF RECLAIMED RUBBER

In view of the variable and complicated composition of reclaimed rubber there is no direct test whereby its presence in a rubber product can be ascertained with certainty, nor is there any procedure of analysis by means of which the proportion of reclaimed rubber can be determined accurately. The presence of reclaimed rubber in a rubber compound is usually inferred from circumstantial rather than direct evidence, consideration being given to physical properties, as well as to analytical determinations.



The physical properties of reclaimed rubber and the respects in which they differ from the properties of new rubber will be discussed in a subsequent section of this circular.

The analytical determinations commonly employed in examining rubber products for reclaimed rubber are acetone extract, combined sulphur, and fillers. A relatively high acetone extract may be due to softeners from reclaimed rubber. However, softeners may be used liberally in some compounds made from all new rubber, and furthermore the softeners employed in reclaiming in some instances may be acidic in nature, and consequently are apt to be present as acetone-insoluble sodium salts.

A percentage of combined sulphur higher than normally required for vulcanization is evidence of the presence of reclaimed rubber because a product made from reclaimed rubber contains the sulphur of vulcanization of the original scrap rubber in addition to the sulphur required for subsequent vulcanization.

TABLE 2.—*Reclaimed rubber*  
[New York quotations, February 24, 1931]<sup>1</sup>

Class	Variety	Specific gravity	Price per pound
High tensile.....	{Super-reclaim, black.....	1.20	\$0.07½ to \$0.07¾
	{Super-reclaim, red.....	1.20	
Auto tire.....	{Black.....	1.21	.07 to .06½
	{Black selected tires.....	1.18	
	{Dark gray.....	1.35	
	{White.....	1.40	
Shoe.....	{Unwashed.....	1.60	.06 to .06½
	{Washed.....	1.50	
Tube.....	{No. 1.....	1.00	.08½ to .08½
	{No. 2.....	1.10	
Truck tire.....	{Truck tire, heavy gravity.....	1.55	.06 to .06½
	{Truck tire, light gravity.....	1.40	
Miscellaneous.....	{Mechanical blends.....	1.60	.05 to .05½

<sup>1</sup> India Rubber World, 83, No. 6, p. 103; Mar. 1, 1931.

The kind and proportion of fillers in a rubber product may give indication of the use of reclaimed rubber; for instance, when a number of fillers for which no specific function is evident are present in small percentages in a rubber product they are more apt to have come from reclaimed rubber than to have been included in the formula for a product made from new rubber. Further, when relatively expensive fillers, such as zinc oxide or antimony sulphide, are present to an extent greater than required by the nature of the product, they are likely to have originated from reclaimed rubber.

## VI. CLASSIFICATION OF RECLAIMED RUBBER

Reclaimed rubber is classified primarily on the basis of the kind of scrap, such as tires, shoes, inner tubes, and the like, from which it was prepared. The main classes are subdivided on considerations of quality, tensile strength, color, and specific gravity. The current classification of reclaimed rubber and the relative value of the different classes and grades are shown by the New York market quotation of February 24, 1931, which is given in Table 2.

It may be noted that the prices, in a general way, are inversely proportional to the specific gravity. This is to be expected since the

grades of lower specific gravity contain more rubber and less filler. Reclaimed rubber of high tensile strength commands a premium as do also light-colored reclaimed rubbers.

High-tensile reclaimed rubber is usually made from inner tubes. It is frequently compounded by the reclaimer with carbon black, zinc oxide, and other ingredients designed to improve the strength and uniformity. This, as well as other kinds of reclaimed rubber that have been made from selected scrap or that have been specially blended or compounded, are sometimes sold under trade brands. The advertising claims usually relate to uniformity, high quality, and special adaptation to particular purposes.

The tensile strength of reclaimed rubber, for purposes of classification and evaluation, is determined on the material after vulcanization. It is common practice to add 5 per cent of sulphur to the reclaimed rubber and to vulcanize specimens of the mixture for different intervals of time. Tensile strength measurements are then made on the various specimens and the maximum value is taken as characteristic of the reclaimed rubber under examination.

## VII. PROPERTIES OF RECLAIMED RUBBER

Some of the properties of reclaimed rubber have already been mentioned in connection with the composition and classification. From a practical point of view the properties fall into two categories, those relating to processes of manufacture and those relating to the serviceability of the products in which reclaimed rubber is used.

### 1. RECLAIMED RUBBER IN MANUFACTURING PROCESSES

Reclaimed rubber finds favor with the manufacturer not only because it is a raw material which is normally cheaper than crude rubber, but also because of the facility with which it can be put through manufacturing processes. Reclaimed rubber is softer than crude rubber, has a lesser degree of elastic recovery, and is particularly well adapted to operations, such as sheeting, tubing, and molding.

Rubber products can be made from reclaimed rubber or from a mixture of reclaimed and crude rubber more quickly, with lower power consumption, and with less waste than with the use of new rubber alone. It is for these and similar reasons that reclaimed rubber may be used in considerable quantity even at a price approximately the same as of new rubber.

### 2. RECLAIMED RUBBER IN RUBBER PRODUCTS

Reclaimed rubber is not equal to new rubber in strength, stretch, or resistance to abrasive wear. In aging qualities reclaimed rubber has long been regarded as inferior to new rubber, but this is not necessarily the case. In softness, flexibility, and related characteristics, reclaimed rubber may compare favorably with new rubber.

#### (a) TENSILE STRENGTH AND ELONGATION

The tensile strength of reclaimed rubber depends in considerable measure on the kind and quality of the scrap from which it was made. Ordinary whole-tire reclaim when vulcanized with sulphur shows a tensile strength at break of 500 to 1,000 pounds per square inch and an elongation of three to four times the original length. In comparison,

new rubber has a tensile strength of 3,000 to 4,000 pounds per square inch, with an elongation of six to eight times the original length. High-tensile reclaim occupies a position intermediate between the above, with a tensile strength of 1,500 to 2,000 pounds per square inch when vulcanized with 5 per cent of sulphur. Special grades of reclaimed rubber produced from new scrap of high quality may have tensile properties which approach even more closely those of new rubber. On the other hand, reclaimed rubber, produced from scrap articles which were themselves made from reclaimed rubber, is distinctly inferior to the ordinary grades. Each cycle of reclaiming produces still further degradation in tensile and other properties. In practice this effect of repeated reclaiming is offset by judicious blending of the scrap so as to produce a material which has a reasonable minimum of strength and elasticity.

#### (b) RESISTANCE TO ABRASIVE WEAR

Products made wholly from reclaimed rubber have a much lower resistance to abrasive wear than similar products made from new rubber. When mixtures are employed the wearing qualities are intermediate between the two and depend on the proportion of reclaimed rubber. Abrasion resistance has been studied particularly in connection with the wear of tires. In an investigation conducted by this bureau, tires were made with treads in which different percentages by weight of new rubber were replaced by reclaimed rubber. When these tires were tested on the road it was found that the resistance to wear was lowered roughly in proportion to the quantity of reclaimed rubber used. This investigation was criticized because new rubber was replaced by the same percentage of reclaimed rubber rather than by an amount of reclaimed rubber which contained the same percentage of hydrocarbon, or, in other words, because no deduction was made for fillers and other nonrubber ingredients in the reclaimed rubber. However, subsequent investigations by other laboratories in which new rubber was replaced by reclaimed rubber on a basis of hydrocarbon content also showed that the resistance to wear was reduced by the introduction of the reclaimed rubber. Further reference to the wear of tires will be made in a subsequent discussion of the economics of the use of reclaimed rubber.

#### (c) DETERIORATION WITH AGE

It is not possible to make any sweeping comparison between reclaimed and new rubber with respect to deterioration with age. It has been a matter of experience in the past that products made from reclaimed rubber have deteriorated more rapidly than similar products made with new rubber. However, with up-to-date compounding and manufacturing procedure, rubber goods can be made which have satisfactory aging properties. The main factors contributing to this improvement are the use of organic accelerators and antioxidants, and the greater uniformity and better quality of the reclaimed rubber itself.

For some uses involving exposure to light and weather, reclaimed rubber is regarded as being more durable and hence preferable to new rubber. One such use is for the production of rubberized top coverings for automobiles, which are said to be made almost wholly from reclaimed rubber.

## (d) SOFTNESS AND FLEXIBILITY

Products made from reclaimed rubber are generally softer and more flexible than products of comparable composition made from new rubber. These characteristics depend on the behavior of rubber when subjected to relatively small deformations. There are few published data which relate either to the softness and flexibility of reclaimed rubber in test compounds, or to the comparative behavior of products made from new and reclaimed rubber in regard to these properties. However, it is generally recognized that reclaimed rubber is adequate and well suited for the manufacture of many types of articles in which softness and flexibility are more important than ability to withstand severe wear. For instance, reclaimed rubber is commonly used in the side wall of tires with little if any of the detriment that would accompany its use in the tread.

## VIII. USES OF RECLAIMED RUBBER

A list of products in which reclaimed rubber has been or may be used in some proportion would include nearly all articles manufactured from rubber. However, in general, rubber products for which a high degree of strength or resistance to abrasive wear are required are made largely or wholly from new rubber, while products not designed for conditions of severe mechanical use may be made almost, if not entirely, from reclaimed rubber.

## 1. PRODUCTS CONTAINING SMALL PERCENTAGES OF RECLAIMED RUBBER

No reclaimed rubber is ordinarily used in the production of dipped goods, such as surgeons' gloves, bathing caps, and toy balloons, or in making gum rubber specialties, transparent sheeting, and the like. Rubber thread, elastic bands, and crêpe soles are other familiar illustrations of products commonly made from new rubber without the admixture of reclaimed rubber.

Relatively little, if any, reclaimed rubber is employed in the manufacture of automobile inner tubes, and only a small proportion ordinarily enters into the composition of the tread of tires. However, when crude rubber is high in price larger amounts of reclaimed rubber may be employed in the tread for the purpose of reducing the cost of the tire. A considerable amount of reclaimed rubber is normally used in the side wall, but the recent vogue for white side walls is said to have caused several manufacturers to use less reclaimed rubber than formerly because reclaimed rubber is suitable only for dark-colored side walls.

The higher grades of electrical insulation and the jacket on portable electric cord are usually made largely from new rubber, though the practice in this regard may vary with the manufacturer and with the specifications under which the material is to be sold.

## 2. PRODUCTS CONTAINING LARGE PERCENTAGES OF RECLAIMED RUBBER

In mechanical goods reclaimed rubber is used in large proportion; in some cases no new rubber whatever is employed. Mechanical goods include such products as gaskets, packings, belting, flooring, heels,

stoppers, and hose. Some grades of electrical insulation such as that employed for the familiar "underwriters' code wire," are made from reclaimed rubber.

Rubberized fabrics are frequently made with a large proportion of reclaimed rubber, though some grades of hospital sheeting are made from new rubber, and balloon and gas-cell fabrics are made exclusively from new rubber.

The manufacture of rubberized fabrics and similar products with latex has its counterpart in the use of dispersions of reclaimed rubber. These aqueous dispersions may be used not only for coating fabric or paper, but also for cementing, binding, and waterproofing purposes.

Rubber footwear, including tennis shoes, boots, and overshoes, is made with a considerable proportion of reclaimed rubber.

Reclaimed rubber is extensively used in the hard-rubber industry, and may be employed in making all but the highest grades of hard rubber or ebonite. In this connection fillers are an important consideration because of their effect on the electrical properties and on the polishing characteristics of the hard rubber. Much use is made of reclaimed rubber having low filler content, such as that produced from inner tubes. The substitution of reclaimed for new rubber in the manufacture of hard rubber has relatively less effect on the mechanical properties than in the case of soft-rubber goods.

### 3. ECONOMICS OF THE USE OF RECLAIMED RUBBER

The primary, though not the exclusive reason for the use of reclaimed rubber is to reduce the cost either of raw materials or of manufacturing processes. Many products, especially among mechanical goods, can be made with the use of reclaimed rubber so as to serve the purpose for which they are intended as satisfactorily as if they were made from all new rubber. In such cases, there is an undoubted advantage in using reclaimed rubber when crude rubber is high priced. However, in case of many other products the introduction of reclaimed rubber reduces the quality and durability at the same time that it decreases the cost. Under some circumstances the reduction in cost may more than offset the reduced serviceability. This may be especially true in times of very high-priced crude rubber.

In the case of automobile tires, several investigations have been conducted to ascertain the relative value of new and reclaimed rubber in terms of tread wear. One such investigation, based on road tests as well as laboratory determinations came to the conclusion that reclaimed rubber at 7 cents per pound cost more per unit of abrasion than new rubber at 20 cents per pound when the two were used together in any proportion. Even with crude rubber at 40 cents per pound reclaimed rubber could be used to advantage in only a limited range of composition. This study did not take into account the costs of manufacture and distribution which would be nearly the same regardless of tire wear. When these factors are considered, any possible advantage to the consumer from the use of reclaimed rubber in the tread of tires seems remote.

TABLE 3.—Consumption of reclaimed rubber in the United States <sup>1</sup>

Year	Reclaimed rubber consumed	Ratio reclaimed to crude	Price of crude rubber	Year	Reclaimed rubber consumed	Ratio reclaimed to crude	Price of crude rubber
	<i>Long tons</i>	<i>Per cent</i>	<i>Cents per pound</i>		<i>Long tons</i>	<i>Per cent</i>	<i>Cents per pound</i>
1917-----	89,168	56.7	72.2	1925-----	137,105	35.3	72.5
1919-----	73,535	36.3	48.7	1926-----	164,500	45.0	48.7
1920-----	75,297	38.4	36.3	1927-----	189,500	50.8	38.1
1921-----	41,351	24.4	16.3	1928-----	223,000	51.0	22.6
1922-----	54,458	19.2	17.5	1929-----	212,700	45.5	20.6
1923-----	69,534	25.3	29.5	1930-----	153,500	40.7	12.0
1924-----	76,072	25.2	26.2				

<sup>1</sup> From Rubber and its Use in the United States, by E. G. Holt, Special Circular No. 1111, Department of Commerce, Bureau of Foreign and Domestic Commerce.

#### 4. SPECIFICATIONS RELATING TO RECLAIMED RUBBER IN RUBBER PRODUCTS

Many specifications for rubber products prohibit the use of reclaimed rubber on the supposition or assumption that goods made with it are inferior to those made with new rubber. In some instances such an assumption is well founded, as in case of products designed for resistance to abrasive wear, or for certain types of electrical insulation. However, in many other instances the use of reclaimed rubber may be amply justified either by the fact that it gives a product as satisfactory for a specified purpose as new rubber or by reason of a saving in cost more than enough to offset the somewhat reduced or impaired serviceability.

In general, the attitude in specification writing toward reclaimed rubber is changing. Two factors are contributing to this change. One is the recent improvement in quality and uniformity of products made from reclaimed rubber, and the other is the development and widespread use of accelerated aging tests which enable the consumer to make comparative estimates of the probable life of rubber products. Before the advent of aging tests new rubber was frequently specified on account of the uncertainty as to the aging qualities of reclaimed rubber. Now new rubber no longer has the benefit of the doubt, but is brought into competition with reclaimed rubber on a performance basis.

#### 5. CONSUMPTION OF RECLAIMED RUBBER

The consumption of reclaimed rubber has undergone wide fluctuations during the recent growth and expansion of the rubber industry. The amount used has evidently been determined in considerable measure by the price of crude rubber. In Table 3 the reclaimed rubber that has been used in the United States since 1917 is shown both in quantity and in proportion to the new or crude rubber consumed. The average price of crude rubber for each year is also shown. In 1917 when crude rubber was 72 cents per pound, the consumption of reclaimed rubber was 57 per cent of that of new rubber, but in 1922, following a drop in the price of new rubber to 16 cents per pound, the consumption declined to 19 per cent of that of new rubber. The price of crude rubber rose to 49 cents in 1926, and by the following year the consumption of reclaimed rubber had mounted to 51 per cent. When the price of crude rubber again

decreased in 1928, 1929, and 1930, the decline in the use of reclaimed rubber was only from 51 to 40 per cent. This decline is far less than might have been predicted from previous experience, especially in view of the fact that the average price of new rubber during 1930 was only 12 cents per pound. The sustained use of reclaimed rubber during the present period (1930-31) of very cheap crude rubber is rendered possible by improvements in the quality and methods of utilizing reclaimed rubber, although other considerations of a different nature are also operative. Manufacturers are reluctant to change their processes and formulas to conform to an apparently temporary depression in the price of crude rubber. Furthermore, the majority of reclaiming plants are affiliated with rubber manufacturing companies who consider it advantageous to continue the production and use of reclaimed rubber even at a loss rather than to shut down the reclaiming plants and permit them to lie idle.

In making comparisons such as the above between the consumption of new and reclaimed rubber it should be borne in mind that a ton of reclaimed rubber is not strictly comparable to a ton of crude rubber. When allowance is made for fillers and other constituents present in reclaimed rubber it is equivalent to only about 60 per cent as much new rubber, on the average.

#### IX. RECLAIMING OF RUBBER ON A SMALL SCALE

Inquiries are frequently received by this bureau from individuals who wish to undertake the reclaiming of rubber on a small scale with the use of simple, inexpensive equipment. However, the processes now in use by the industry are not adapted to small-scale operation. A large outlay is needed for machinery and equipment, as might be inferred from the description of reclaiming processes given in an earlier section of this circular.

Not only is special equipment required, but skilled technical control and supervision is equally essential. The successful operation of a reclaiming plant requires the services of engineers, chemists, experienced operators, and laborers. So far as is known no publication is available which purports to give instructions for assembling and installing equipment for reclaiming, or directions for operating a reclaiming plant.

The market price of reclaimed rubber is relatively low, and although the price fluctuates somewhat there is seldom, if ever, a large margin of profit. Consequently, in order to be economically practicable, reclaiming must be conducted on a large scale, particularly in view of the large outlay required for equipment, and the overhead entailed by the necessary technical control and supervision.

A more practical means than reclaiming for the small-scale utilization of waste rubber products is to employ them in the direct manufacture of other rubber products. In the case of discarded tires, for instance, much of the rubber and fabric may be strong and capable of additional wear, although the tire as a whole is unserviceable. Accordingly, a variety of smaller articles may be fashioned from portions of the tire. In many parts of Asia and Africa sandals and soles of shoes are cut from discarded tires. This work is usually done by hand, and the product, though very durable, is often crude according to western standards. In the United States tires are not utilized in

this way, though discarded belting is said to be so employed to a limited extent. Discarded tires are, however, regularly employed in the manufacture of mats; the tires are cut into small blocks or links which are arranged edgewise and in alternate rows and are secured together by galvanized or brass wire. The mats so prepared present a surface of the cut edges of the rubber and fabric of the tire and are not slippery when wet. Mats of this description are used as doormats and as deck mats on shipboard.<sup>5</sup> Discarded tires are also employed for the manufacture of tire boots and blowout patches. Discarded inner tubes are said to be employed to a limited extent for the production of elastic bands, which are cut from the tubes by means of a special machine. Certain kinds of gaskets are made by punching them from the less worn portions of discarded tires and belting. Many other uses for waste-rubber products have been suggested and some of these are being exploited in a small way.

There are a number of patents which relate to direct uses for waste rubber; a list of such patents with a brief abstract of each is given in *India Rubber World*, vol. 77, No. 6, pp. 60 to 62; March, 1928.

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This bibliography is made up chiefly of the publications on reclaimed rubber which have appeared in the United States since 1925. It is not exhaustive and does not include patents. For the convenience of the reader the publications have been classified according to subject matter, though the nature and scope of certain of the publications is such that their classification has been somewhat arbitrary.

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## XI. ACKNOWLEDGMENT

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