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THE MANUFACTURE OF AIRTIGHT LEATHER GASKETS

BY

W. KEDZIE TELLER

A

THESIS

submitted to the faculty of the
SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI
in partial fulfillment of the work required for the

Degree Of
CHEMICAL ENGINEER

Rolla, Mo.

1929.

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Approved by. *W. T. Schrenk*

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THE
MANUFACTURE OF AIRTIGHT LEATHER
GASKETS.

Reasons for Investigating this Field.

Wherever gaskets are used in conjunction with any type of mechanism operated by compressed air, of course, it is essential that the gaskets be air tight.

Whereas it is possible to use rubberized fabric for this purpose with a degree of satisfaction, treated leather has certain advantages which make its use preferable. Leather is readily available and can be cut and molded into various shapes with less difficulty than is encountered in fabricating similar designs from reinforced rubber. Then, too, leather withstands wear much better than rubberized fabric.

The problem then arises to treat leather to make it airtight, for leather as we ordinarily know it is a very porous material. The treatment must also waterproof the leather since it lasts much longer if it is not affected by water.

Gaskets in certain types of mechanisms, such as railway car airbrakes, are subject to temperatures

ranging from 120°F. down to -30°F. and sometimes lower. The ideal leather treatment must be such that it will not cause the gaskets to become stiff at cold temperatures nor melt and be forced out in hot weather.

Available Treatments and Attempts
to Improve Them.

At the time this problem was begun available treatments were not satisfactory from the standpoint of the extreme temperatures noted above. In cold weather the leather became so stiff that proper seating of valves was not the rule and on hot days the treatment was often forced out of the pores of the leather under the high air pressure used.

These available treatments were all blends of waxes, fats and greases. Leather was impregnated by immersing it in molten mixtures of these materials.

Believing it possible that a more suitable combination of wax and grease might be found this point was first investigated.

Combinations of ceresin, carnauba and beeswax with various greases similar to vaseline were gone into in detail in an attempt to secure a suitable filler. These mixes were examined for softening

points and brittle points since a treatment which would not fall within the temperature range of -30°F . to 120°F . would not meet the desired specifications. To be more explicit, if a certain mixture of wax and grease softened materially at 120°F . it would be unsuitable and was discarded. Likewise a mix that became brittle at -30°F . was considered useless.

Inasmuch as the attempt to blend these materials brought no better results than some of the available treatments, from the temperature standpoint, it was not found necessary to actually fill any leather for further testing. Later as new products were devised it was found desirable to design and construct a testing machine to prove the airtightness of gaskets treated with these products.

Insoluble Soaps.

The wax and grease idea was finally disposed of as impractical since combinations with suitable softening or melting points were invariably brittle when cooled below zero and vice versa.

Soaps of lead, manganese or calcium seemed to offer a likely field. Accordingly several batches

of these were prepared.

A few typical formulae are indicated:-

(A)
Corn oil fatty acids----- 40 grams
Lead subacetate----- 10 "
Hold at 240°C. for 10 minutes.

The resulting mass was not liquid at 110°F.
and was quite stiff at low temperatures.

(B)
Corn oil fatty acids----- 40 grams
Lead subacetate----- 18 "
Too hard when cooled to +10°F.

(C)
Corn oil fatty acids----- 40 grams
Lead subacetate----- 12 "
Too soft at 120°F.

Several other various similar combinations all
gave unsatisfactory products as did also those made
either with manganese acetate or calcium acetate.

Cellulose Esters.

Solutions of both cellulose acetate and cellulose nitrate were experimented with. The biggest difficulty encountered here was in forcing the solution into the pores of the leather. Even when the leather had been apparently well soaked in such

solutions and then dried the actual amount of ester deposited in the leather was very small. Cellulose ester solutions can not be made concentrated without increasing their viscosity beyond the point where they will penetrate the minute pores of leather. This made thorough impregnation of the gasket impossible since upon evaporation of the solvent there is always a diminution in volume.

Rubber Solutions.

Rubber solutions being extremely viscous were found to fail for reasons similar to those which made cellulose esters unsatisfactory.

Polymerized Oils.

Another possibility appeared to lie in the field of polymerized oils. Although this point was appreciated from the beginning of the investigation, and work was constantly being carried on along this line, success was not assured until after the other methods described had been given up as impractical. Accordingly those experiments which did not lead to successful results will be considered first.

Castor oil when heated with zinc chloride thickens to degrees varying with the time and temperature of heating and the amount of zinc chloride used.

Below are a few of the typical combinations with the treatments given them:-

#1.

Zinc chloride----- 1/2 gram
Castor oil----- 20 grams

Heated at 190-200°C. for 30 minutes.

When cooled this product was not much thicker but when again heated to 200°C. and held there for ten minutes it was noticeably viscous.

#2.

Zinc chloride----- 1 gram
Castor oil----- 20 grams

After heating at 200°C. for 20 minutes this product solidified.

#3.

Zinc chloride----- 0.65 gram
Castor oil----- 20 grams

Held at 200-210°C. for 30 minutes.

This resulted in a slowly flowing viscous liquid. After adding 5 grams of beeswax a solid resulted which was not liquid at 120°F.

#4.

Zinc chloride----- 0.75 gram
Castor oil----- 20 grams

Heated at 210-220°C. for 30 minutes.

10 grams of ceresin was then added and the resulting mass had a melting point above 130°F. A piece of leather was treated with this mix by immersing the leather in the molten mix at 180-190°F. This gave a gasket from which the treatment ran out at 120°F. under 90 pounds per square inch air pressure.

#5.

Zinc chloride----- 0.95 grams
Castor oil----- 20 grams

Heated at 200-210°C. for 30 minutes and then mixed with 15 grams of beeswax. A gasket treated with this mix leaked only enough to drop air pressure from 95# to 65# in 70 minutes.

#6.

Zinc chloride----- 0.95 gram
Castor oil----- 30 grams

Heated at 200-210°C. for 40 minutes when it congealed. An attempt was made to thin this jelly down by heating with mineral oil but with no marked success. Although this combination appeared

to have a future it was not investigated further because of greater success along other lines.

Another oil known to be a quick polymerizing oil is chinese wood oil. By soaking leather in this oil and hastening polymerization with heat it was possible to produce a fair product, but it would not withstand high air pressure - 100# per sq. in.

Sulphonation of this oil was next attempted and below are given some of the experiments:

#1. 25 grams of oil was treated with 12½ grams of 80% sulfuric acid. A spongy rubberlike mass resulted.

#2. 25 grams of oil was treated with 60% sulfuric acid. A sticky mass resulted. This was washed and boiled in 2% ammonia and washed again. It dried very slowly when exposed to the air.

#3. 20 grams of oil was heated with 6 grams of 33% sulfuric acid for 20 minutes at 120°C. The resulting mass was washed and neutralized and found to be a rubberlike solid.

Various further experiments with chinese wood oil all gave products which would have been excellent could they have been carried into the pores of the leather. This fact presented a real difficulty for it made the task appear hopeless unless part of the polymerization could be accomplished after impregnation.

As stated before, diminution in volume through evaporation of a solvent made impregnation with solutions of otherwise satisfactory fillers impractical. The most likely possibility seemed to be in thinning some partially polymerized product, and also retarding polymerization long enough to enable one to impregnate the leather with a material which would later set hard enough and still fill all pores.

Sulfurized Corn Oil.

Throughout the investigation sulfurized corn oil experiments seemed to offer most success, but the problem mentioned in the preceding paragraph was always the stumbling block which caused the work to go off on a tangent.

Having worked on "Art gum" and knowing the

method of producing sponges made partially of corn oil the simple corn oil jelly was easily and quickly produced by the following formula:-

Corn oil-----	40 grams
Sulfur-----	10½ "

Heated at 150°C. for about 2 hours this combination developed into a very tough rubberlike product. This material did not become brittle nor even hard at -40°F. nor soften appreciably at 150°F.

Its solubility in various organic solvents was next studied. Benzol, carbon bisulfide, chloroform and carbon tetrachloride all had little if any solvent action. Petroleum ether, gasoline and naphtha all appeared to be good solvents but worked slowly.

The idea of adding a retarder to the above formula so that the point at which polymerization takes place could be controlled was next studied. As made following the above formula the mixture was a very thin liquid up almost to the minute it jellied, and the solidifying took place almost instantaneously. If this jelling could be slowed down indefinitely, once it had started, and then caused to proceed at will of the operator great possibilities might be

expected.

Since the rubberlike sulfur-cornoil dissolved in petroleum fractions, various other petroleum products which were nonvolatile at 150°C. (the temperature used for polymerizing) were added in varying amounts to the original corn oil and sulfur batch before heating. These batches were then heated for the customary two hours or longer if necessary at 150°C. until the mixes stiffened perceptibly.

Using a medium grade lubricating oil as a retarder it was found possible to control polymerization. The following formula gave a mix which when almost gelatinized could be cooled down below the boiling point of naphtha, and then mixed with naphtha to give solutions which were not viscous yet were fairly concentrated. By later evaporating off the solvent a rubberlike solid resulted having the desired properties for filling the pores of leather.

Corn oil-----	40 parts
Medium lubricating oil---	15 "
Sulfur-----	10.5 "

This mix was heated at 150°C. with continual stirring until gelatinization just commenced. This required from 3 to 5 hours, the time seeming to vary more with small batches than with large. The larger

batches usually required a longer cooking than the smaller.

When the point of incipient gelatinization was reached the batch was cooled down to about 110-120°C. and mixed quickly with 40 parts by weight of naphtha. The resulting liquid was used for impregnating the leather.

The dry leather was immersed in this liquid and soaked for one hour at a temperature not exceeding 120°C. It was then removed and allowed to hang at room temperature until the surplus filler drained off. The remaining filler was then squeezed off and the gasket placed in an oven and kept at 90°C. for 24 hours. A further storing at room temperature for two weeks before using gave the best results.

Tests for airtightness were conducted on the testing apparatus shown in plate which was designed and constructed specifically for this purpose by the author.

This method of treatment gave a gasket which remained airtight and waterproof as well as pliable and serviceable throughout a temperature range of -40°F. to +140°F.

The development of this process for treating

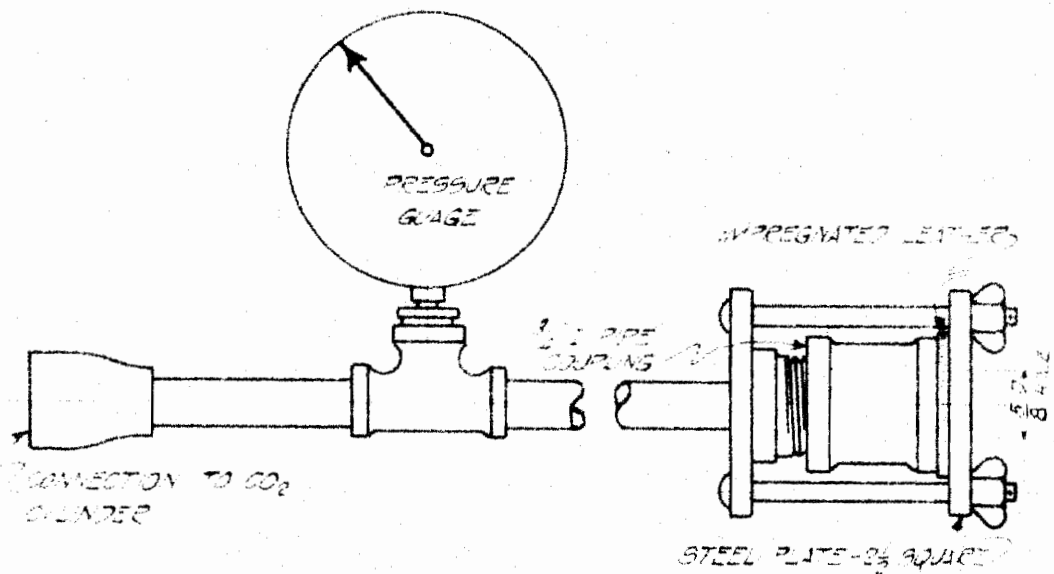
leather did not involve what might be termed intricate chemical research, but rather a common sense application of scientific and practical knowledge acquired through contact with many varied problems encountered in several years' experience in industrial chemistry.

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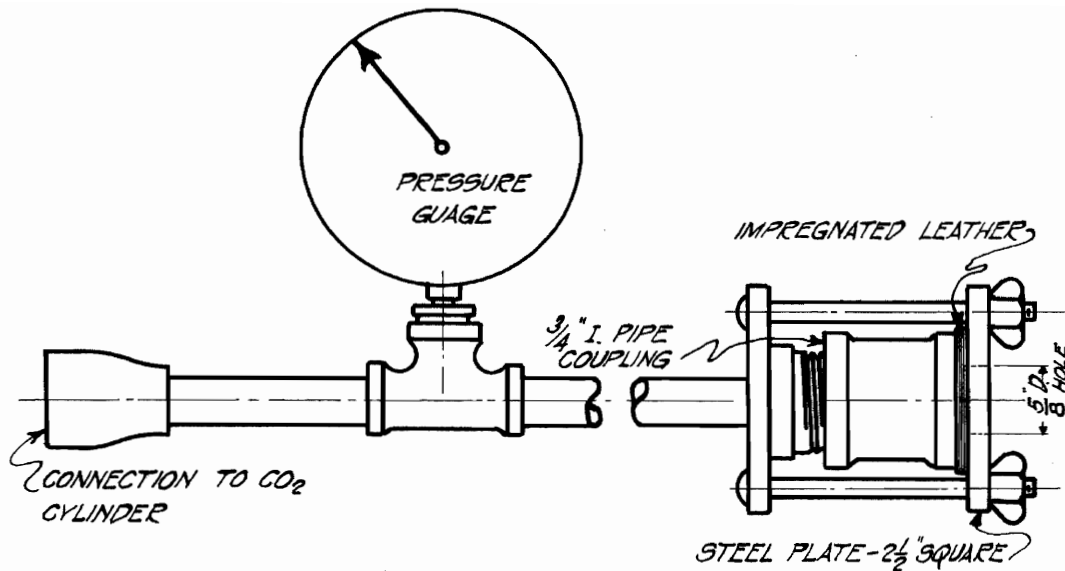
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THE IMPREGNATED LEATHER IS SHOWN CLAMPED BETWEEN THE IRON PIPE COUPLING & THE STEEL PLATE. THE PLATE HAS AN OPENING CONCENTRIC WITH THE OPENING IN THE COUPLING WHICH PERMITS AIR OR GAS TO PASS THRU A LEAKY PIECE OF IMPREGNATED LEATHER.

THIS APPARATUS MAKES IT EASILY POSSIBLE TO TEST TREATED LEATHER UNDER VARIOUS TEMPERATURES BY PLACING THE PORTION TO THE RIGHT INSIDE OF A THERMO-REGULATED OVEN.

~ SKETCH OF TESTING APPARATUS ~



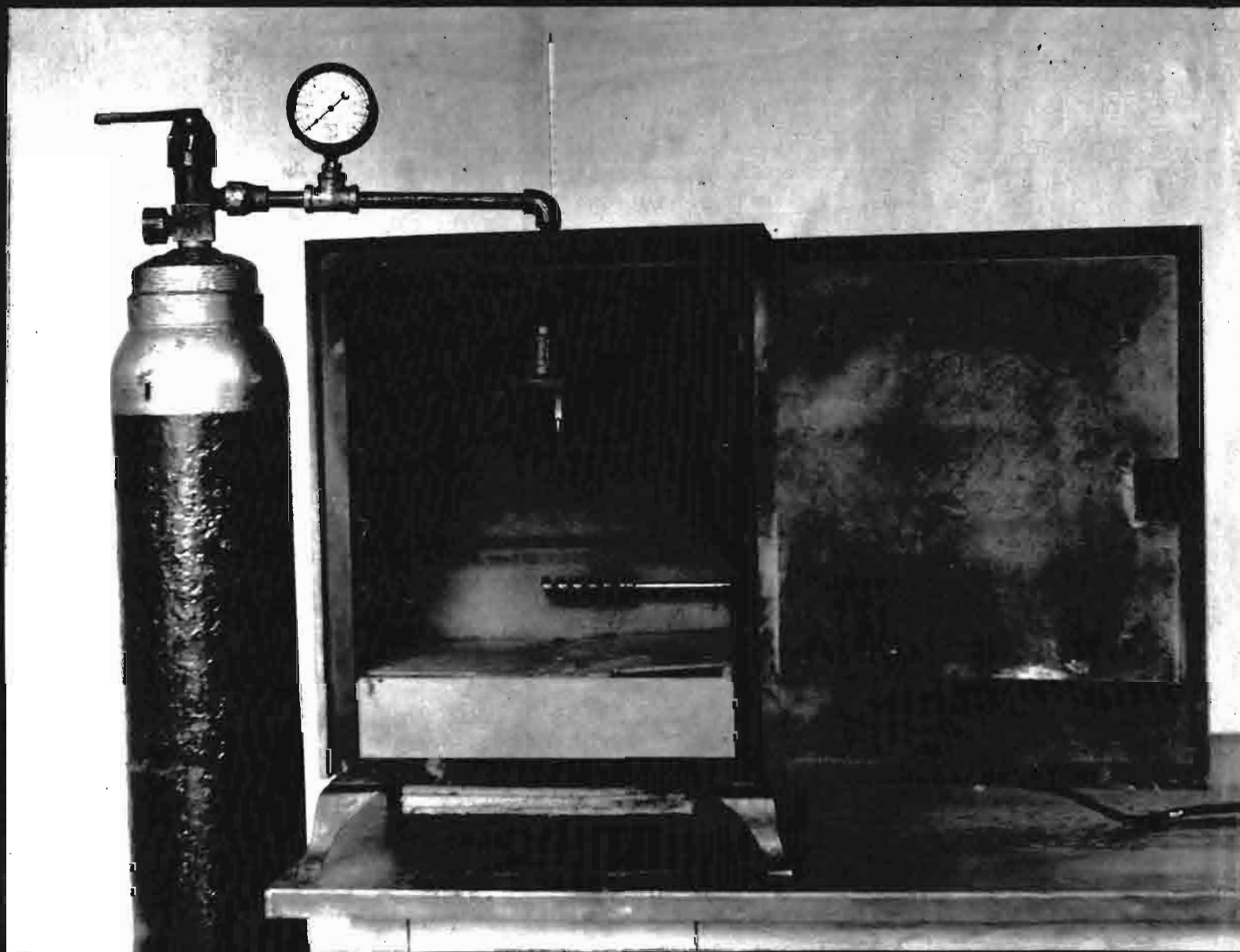
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Apparatus Used in
Testing Gaskets for Airtightness



Apparatus as Used for Testing Gaskets at a Temperature of 130°F.