Development of ENR Based Compounds for Rice Dehusking Rubber Rollers

(Carried out at R R I I, Kottayam)

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By :
JATINDER PAUL

Guides :

MR. VINAYAK M. HEGDE
M-Tech, M.I.S.T.E.,
Lecturer in Chem. Engg Dept.

DR. N.M. MATHEW
Joint Director (RT)
R R I I, Kottayam.

Department of Chemical Engineering

SIDDAGANGA INSTITUTE OF TECHNOLOGY
TUMKUR - 572 103
KARNATAKA
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CHAPTER - 1

INTRODUCTION

1.1 GENERAL INTRODUCTION

Rubber covered rollers are important production tools in many of industries. These cover a wide field ranging from typewriter to textile industry. A rubber roller is in fact a cylinder of rubber bonded to a rigid core which is usually made of metal. The functions of rubber rollers are manifold depending on the widely varied uses to which rolls are put. These are employed in varied occupations such as cracking nut shells, squeezing seeds out of resins, pulling hairs out of hides and dozen of other operations in the textile, food processing, metal, leather, glass, plywood and other industries. [1,2]

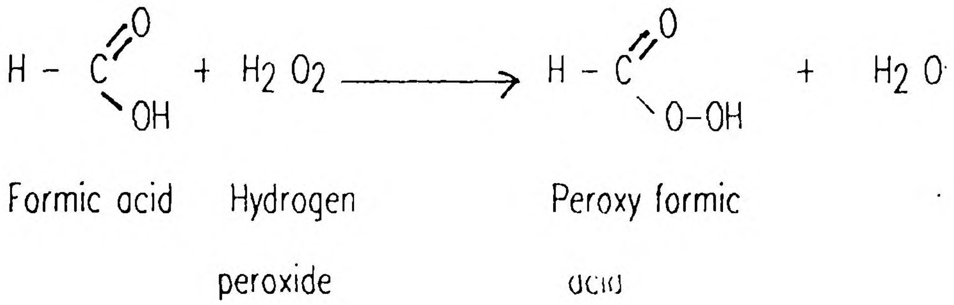
The reasons for such widespread use of rubber for roll covering does not lie in any single quality, unless versatility can be described as a quality. Rubber compound for rolls can be made as hard as bone or as soft as gumdrop.[1,2] The suitability of a rubber roller for any particular application will mainly depend on the merit of the rubber compound to meet the service requirements. Thus proper designing of the rubber compound is of utmost importance in the manufacture of rubber rolls.

1.2 EPOXIDISED NATURAL RUBBER (ENR
- CHEMISTRY AND PRODUCTION

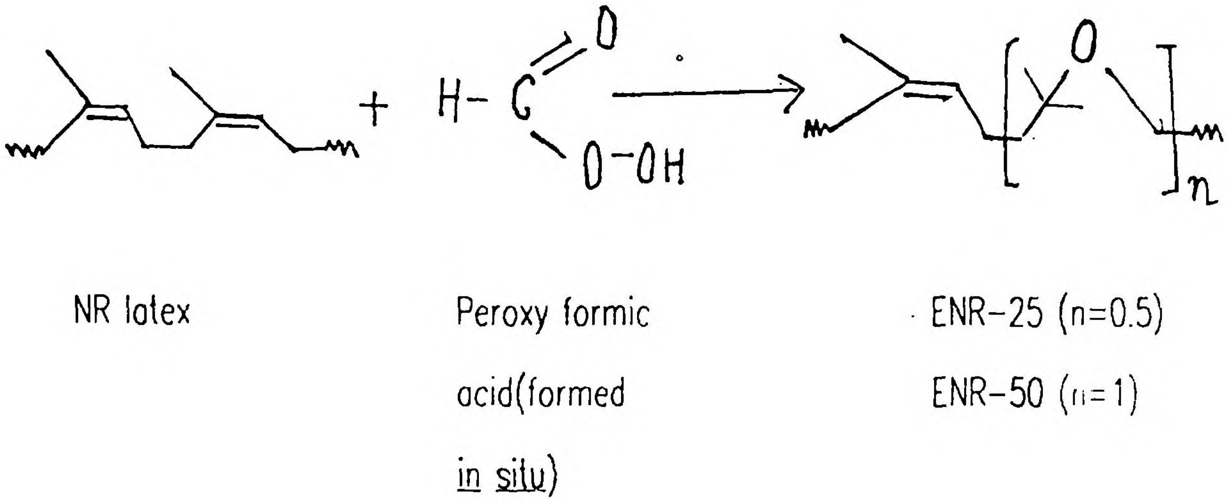
The mechanical properties of natural rubber (NR) are generally superior those of synthetic rubbers. However NR can not compete with speciality synthe elastomers with regard to such properties as gas impermeability and oil resistanc This led to the development of epoxidised natural rubber (ENR). ENR is derived chemical modification of NR, which converts the unsaturation in the rubber in epoxide groups. This new polymer has improved oil resistance and lower ga permeability, whilst retaining many of the inherent properties of NR such as stra crystallization and also exhibiting some novel features.[3,4]

PRODUCTION OF ENR.

All grades of ENR are prepared from premium quality NR latex employing the hydrogen peroxide/formic acid route formed in situ (same place). The latex is first stabilized against acid coagulation by the addition of a non-ionic surfactant and expoxidation is carried out at about 60°C. The reaction conditions are carefully controlled to avoid secondary ring opening reactions. It has been reported that under carefully controlled conditions NR latex can be epoxidised to over 75 mole%without the formation of secondary ring opened structure. The epoxidised latex is neutralized prior to heat coagulation and thoroughly washed before drying in hot air. At present ENR 25 and ENR - 50 are two important commercial grades of ENR. In ENR - 25, 25 mole % of the unsaturation is converted to epoxide group and in ENR - 50, 50 mole % unsaturation is converted into epoxide group.[4,5] The reactions are described below.



(a) Peroxy formic acid formed in situ.



(b) Production of epoxidised natural rubber.

1.3 PROSPECTS FOR RICE DEHUSKING RUBBER ROLLERS

The demand and supply of rice dehusking rubber roller is directly related to rice production. There was an increasing trend in rice production during 1989-90, 90-91, 92-93 and 1993- 94. It is estimated that rice production will be 88 million MT by 1996-97 and by the turn of the century it will be about 100 to 105 million MT, therefore manufacturers of rice dehusking rubber rollers can expect a growing market in the years to come.[5]

PRODUCTION OF RICE RUBBER ROLLERS (in pieces.) [6]

YEAR	1989-90	1990-91	% GROWTH	1991-92	% GROWTH	1992 - 93	% GROWTH
RUBBER ROLLERS.	186400	217965	17.0	211043	-3.2	215960	2.3

Annual average rate of growth is recorded as 5.4 percent during 1989-90 to 1992 - 93 period for production of rice rubber rollers.

The increase in export of rice from India is due to the international acceptability of its quality which is due to the result of modernization of rice mills, and this offers a better scope for manufacturers of rice rubber rollers. Rice rubber rollers have to be replaced often after required service life, the market potential of the other countries is still to be exploited. All these considerations show bright future for rubber rollers for rice dehusking.

1.4 AIM AND OBJECTIVE OF THE WORK

Due to modernization of rice mills, rice dehusking rubber rollers have gained importance in recent years in the agricultural sector. This improved process is now increasingly being used in place of the conventional mill in which the rice is processed by custom or cooling system is employed. The major advantages of modern rice mills is that the new system does not require cooling and yields better quality rice (whole rice with minimum percentage of broken rice) at greater efficiency and low operational costs.[7,8,9]

At present rollers covered with NR and polybutadiene rubber (BR); nitrile rubber (NBR) and carboxylated nitrite rubber; styrene butadiene rubber (SBR), neoprene rubber and other imported synthetic rubbers or their blends are being used in the manufacture of rice dehusking rubber rollers.[10,11,12]

Epoxidised natural rubber (ENR) is a newly developed chemically modified natural rubber which claims to possess better oil resistance than NR. The factors that lead to the feasibility evaluation of ENR for making rice dehusking rollers are given under.

- Local manufacturers desire to switch over to a 100% indigenous polymer.
- Rising prices of synthetic rubbers and even occasional short supply situation.
- Large and growing market potential.
- Lack of published information.
- Better substitute for NR rollers.

CHAPTER - 2

LITERATURE SURVEY

CHAPTER - 2

LITERATURE SURVEY

2.1 DEFINITIONSOF THE TECHNICAL TERMS USED RELATING TO RUBBER[13,14]

ABRASION: The surface loss of a material due to frictional forces.

ABRASION LOSS: Volume of rubber abraded from a specified test piece under specified conditions.

ACCELERATED AGEING TEST: Test in which an attempt is made to produce the effects of natural ageing in a shorter time and to measure them.

ACCELERATOR (RUBBER): Compounding ingredient used in small proportions to cause vulcanization to take place in a shorter time or at a lower temperature.

ACTIVATOR: Compounding material used in small proportion to increase the effectiveness of an accelerator.

AGEING: Changes which take place in rubber with the passage of time. Qualification of this term is usually necessary, e.g., heat ageing, light ageing.

ANTIOXIDANT : Compounding ingredient used to retard deterioration caused by oxidation.

BLOOM: Material which has diffused to the surface of rubber

COMPOUND: An intimate admixture of a polymer(s) with all the materials necessary for the finished article.

COMPOUNDING INGREDIENT: Material or substance added to a rubber to form a mix.

COMPRESSION MOULDING: Moulding process in which the blank is placed directly in the mould cavity and compressed to shape by close of the mould.

COMPRESSION MOULD: Mould for compression moulding.

COMPRESSION SET : The residual deformation of a material after removal of the compressive stress.

CONVENTIONAL SULFUR VULCANIZING SYSTEM: As applied to natural rubber and isoprene and butadiene based synthetic rubbers, a vulcanizing system using a relatively high proportion of free sulphur and producing at optimum cure a network in which the combined sulphur exists predominantly in the polysulphidic crosslinks and in non- crosslink chain modification.

CROSSLINK: Chemical bond bridging one polymer chain to another.

CROSSLINKING : Formation of chemical bonds between polymer chains to give a network structure.

CURE : Vulcanization conditions, necessary to produce a given state of vulcanization.

CURING AGENT : Synonym for vulcanizing agent, which is the preferred term.

DUMBBELL SPECIMEN (RUBBER) : A flat specimen having a narrow straight central portion of essentially uniform cross section with enlarged ends.

DUROMETER : An instrument for measuring the indentation hardness of rubber.

EFFICIENT VULCANIZATING (EV) SYSTEMS : As applied to natural rubber and isoprene and butadiene based synthetic rubbers, a vulcanizing system making efficient use of sulphur and producing at optimum cure a network containing a preponderance of thermally stable monosulphidic cross links.

ELASTOMER : A term often used for rubber and polymers that have properties similar to those of rubber.

ELONGATION : Extension produced by a tensile stress.

ELONGATION, (PERCENT) : The extension of a uniform section of a specimen expressed as percent of the original length.

FLASH : Surplus material forced from a mould on closure under pressure.

FORMULATION : A list of the materials and their amount used in the preparation of a compound.

FRICTION RATIO : Ratio of surface speeds of two adjacent rolls (mills, callendar or refiner).

HARDNESS : The resistance to indentation as measured under specified conditions.

HEAT BUILD UP : The accumulation of thermal energy generated within a material as a result of hysteresis, evidenced by an increase in temperature.

HYSTERESIS : Visco-elastic effectby which mechanical energy is irreversibly converted into heat during cyclic deformation.

INHIBITOR : A material used to suppress a chemical reaction.

INTERNAL MIXER : Mixer consisting of specially shaped rotors operated in a closed chamber.

MASTER BATCH : A homogeneous mixture of rubber and one or material in known proportions for use as a raw material in the preparation of the final compounds.

MASTICATION : A breakdown or softening of raw rubber, or a mix by the combined action of mechanical work (shear) and atmospheric oxygen, sometimes accelerated by the use of a peptizer and frequently at elevated temperatures.

MILL : A machine with two counter rotating rolls, usually driven at different speeds, having adjustable longitudinal axis separation from one another to alter the nip, used for breakdown mastication, mixing or sheeting.

MODULUS : Tensile stress required to stretch the test piece from the unstrained condition to the given elongation.

MOONEY VISCOSITY : Measure of the viscosity of a rubber determined in a certain shearing disc viscometer.

NATURAL RUBBER : A cis -1,4-polyisoprene that is obtained from a botanical source, usually hevea brasiliensis.

OPTIMUM CURE : The state of vulcanization at which a desired property value or combination of property values is obtained.

PLASTICITY : A characteristic of unvulcanized rubber indicated by the degree of retention of deformation after removal of the deforming force.

PLASTICIZER : A compounding material used to enhance the deformability of a polymeric compound.

PROCESSABILITY : The relative ease with which raw or compounded rubber can be handled in rubber machinery.

PROCESSING AID : A compounding material that improves the processability of a polymeric compound.

REINFORCEMENT : The act of increasing the mechanical performance capability of a rubber by the incorporation of materials that do not participate significantly in the vulcanization process.

REINFORCING AGENT : A material, not basically involved in the vulcanization process, used in rubber to increase the resistance of the vulcanize to mechanical forces.

RESIN : An organic material of indefinite and relatively high molecular mass which may be used as a softener, processing aid, Vulcanizating agent, or reinforcing agent.

ROLL : Roller or hollow cylinder forming a major moving member of a rubber processing machine.

SEMI - EFFICIENT VULCANIZING (semi -EV) SYSTEMS : As applied to natural rubber and, isoprene and butadiene based synthetic rubber, a vulcanizing system having sulphur and accelerator concentration between those of a conventional sulphur and EV systems.

SET : Strain remaining after complete release of the force producing the deformation.

SPECIMEN : A piece of material appropriately shaped and prepared so that it is ready to use for a test.

SYNTHETIC RUBBER : Rubber produced by polymerizing one or more monomerswith or without post-polymerization chemical modification.

TEAR STRENGTH : The maximum force required to tear a specified specimen, the force acting substantially parallel to the major axis of the test specimen.

TENSILE STRENGTH : The maximum tensile stress applied, during stretching a specimen to rupture.

VULCANIZATE : The product of vulcaniztion; a crosslinked rubber.

VULCANIZATION : An irreversible process during which a rubber compound, through a change in its chemical structure (for example, cross-linking), becomes less plastic and more resistant to swelling by organic liquids while elastic properties are conferred, improves, or extended over a greater range of temperature.

VULCANIZING SYSTEM : The combination of vulcanizing agent and, as required accelerators, activators, and retarders used to produce the desired vulcanization characteristics or vulcanizate characteristics.

2.2 RICE MILLING

The rice kernel is covered with a highly adhering hull which is relatively high in silicon, thus rendering it unsuitable for consumption. To convert paddy rice into the marketable form dehusking has to be carried out. In the past, it was done manually by pounding the paddy contained in a receptable with a wooden stump, which is still practiced in certain parts of our country and was once part and parcel of village life. Because of the rapid stride in industrialization, hand pounding was replaced by mechanical dehusking with stone or emery - lined grinders. [7,8,9]

The seriousness of the food problem and to cope up with such a compelling necessity, it has become necessary to study all the aspects of improvements in milling system. This gave birth to the modern rice milling industry as it stands today. In most of the rice growing areas it is one of the largest single food industry.[9]

In general, in the rice industry, when one refers to a modern rice mill it includes series of processing operations. The units in a modernized rice mill are shown in Figure 1.[9] The modern rice milling installations have basically pre cleaning devices, shellers, paddy separators and polishers. Other units are optional and depend upon marketing management or other considerations. The modern processing of rice consists of essentially the same steps.[7,8]

1. Cleaning the incoming rough paddy.
2. Shelling the rough paddy.
3. Milling to remove the bran from brown rice.
4. Grading the milled rice by length into whole grain and brokens.
5. Mixing milled grains and brokens to meet specification of buyers.
6. Packaging.

2.3 RUBBER ROLLER SHELLER

2.3.1 ADVANTAGES

Paddy dehusking by rubber roller sheller is now increasingly being used in place of the conventional use of emery shells. This type of machine; which appears to have originated in Japan is becoming increasingly popular and is known by many different names such as shellers, hullers, huskers, dehuskers, etc.[8]

The switch over from conventional shelling to rubber roller dehuskers was because of the resilient nature of the rubber rolls, other advantages being[7,8,9]

1. It does not require cooling system.
2. Yield of husked rice is high.
3. Breakage rate is low during dehusking operation.

4. Channelize the by-products of paddy like bran and husk for better utilization.
5. White coloured finish rice with no blackishness.
6. Changing roll is simple and they can be retreaded.
7. Greater efficiency at low cost.

Due to these advantages, the rubber roller sheller is considered the best among the shelling equipments and it is now widely used throughout the world.

2.3.2 WORKING

Before designing a compound for a good paddy dehusking roller, it is useful to examine briefly the working of rubber roller sheller.

The first step in the actual milling of rice is to remove the shells ie., shelling. A crosssectional side view of apaddy dehusking rubber roller sheller is illustrated in Figure 2. These are essentially a pair of rubber rolls which are turning in opposite direction and at different speeds, through which paddy is passed on upper side. Paddy runs between the rollers in a thin even sheet, a shearing action is created on paddy by contact with the rubber rollers operating with a differential displacement action, which strips off the husk from the grain. Paddy feed is regulated by an admission shutter. A regulating wheel makes it possible to control the pressure on the paddy between the rollers. An enlarged view at point 'A' of Figure 2. is also shown for clarity. The product of the sheller is a mixture of whole grain brown rice, broken brown rice, unshelled rough rice and hulls. In practice the dehusking efficiency can be as high as 90% per pass.

2.4 TYPES OF RUBBER ROLLERS

There are two types of rollers being used in modern rice mills. According to Indian Standards IS-8427-1982 they are

a. Type A - Rolls of black colour.
b. Type B - Rolls of white or any colour other than black.

Both types mentioned shall be either key type as shown in Figure 3. or slip-on type as shown in Figure 4. The keys may be rectangular or taper.

The dimensions of these rollers vary depending on their application in different types of mills. The quality of the rollers are estimated from the durability of these rollers which depends on output capacity of rice rubber roller, their sizes as well as their uses i.e., whether the rollers are used for raw milling or for parboiled rice milling. This can be assumed by the quantity of paddy that can be shelled under normal conditions by using a pair of rollers in a particular mill.

2.5 OPERATING CONDITIONS FOR RUBBER ROLLER SHELLER

Paddy husk is a very abrasive material, high in silica content with a hardness of 6½ on the Mohs scale. While in operation rice mills are often run between 15 to 20 hr. a day nonstop at 1.28:1 friction ratio with the faster roller at 900 rpm. Roller temperature may reach upto 80°C which leads to a rapid wear of rollers. These rollers are also acted upon by rice bran oil during dehusking operation.

Taking these factors into account a paddy dehusking roller should satisfy the following properties to withstand the critical operating conditions. [10,11,12]

- High hardness.
- Excellent heat and abrasion resistance.
- Good service life.
- Rice bran oil resistance.
- Non-toxic, since used for food stuffs.
- Should obtain good finish product and white rice.

- The surface must be smooth and absolutely free.
- It should have the correct degree of resilience.
- Low cost.

2.6 EXISTING LITERATURE

So far no work has been reported on development of ENR based compound for rice dehusking rollers. Formulations based on NR, NBR, SBR and blends of different synthetic rubbers for rice dehusking rubber rollers are available in product profile of different synthetic rubber manufacturing companies. Project work and evaluation studies has also been carried out for these rice dehusking rubber roller shellers.[11,12]

2.7 IS SPECIFICATIONS (IS 8427-1989)

MAIN FEATURES

TYPES :-

The rubber rolls shall be of the following two types.

a. Type A - Rolls of black colour

b. Type B - Rolls of white or any colour other than black.

PHYSICAL PROPERTIES :-

1. TENSILE STRENGTH :- Shall be minimum of 11.8 MPa (Mega pascal) and 8.8 MPa for Type A and B rolls respectively.
2. ELONGATION :- Shall be minimum of 150 percent and 130 percent for Type A and B rolls respectively.
3. HARDNESS :- For both the types of rubber rolls shall be 85 to 97 IRHD (International Rubber Hardness Degree.).

OTHER REQUIREMENTS

1. The rubber rolls shall be uniform in construction.
2. The rubber surface shall be free from sulphur bloom°.
3. The rolls shall be free from blisters, pin holes, cracks, embedded foreign matter, air bubbles and other defects which may impair their serviceability.

2.8 SELECTION OF COMPOUNDING INGREDIENTS

Rubber compounding is the art and science of selecting and combining elastomers and additives to obtain an intimate mixture that will develop the necessary physical and chemical properties for a finished product. The mixture of rubber and compounding ingredients is known as a compound. The principal task of compounding is to secure an acceptable balance among properties, processability and price.

Rubber compound is developed from the following ingredients.[10,15,16,17,18,19,20]

1. Base polymer or blend of polymers
2. Curatives
    a. Curing agent
    b. Accelerator
    c. Activator
3. Aids to processing
4. Aids to quality
    a. Fillers-reinforcing type
    b. Antioxidants
5. Special materials

The various materials used in compounds and their functional class are given in Table 1.

For the present work the following ingredients have been selected for the product development to ensure service properties requirement.

2.8.1 BASE POLYMER OR BLEND OF POLYMERS

The base polymer or the blend of polymers are to be selected according to the broad pattern of final properties required. NR is selected because of its mechanical properties and easy availability. NBR is chosen as control compound which is being used presently for these rollers. It has good physical properties , better ageing resistance and excellent rice bran oil resistance. ENR-50 was chosen as test material for product development as it claims to have oil resistance property while retaining mechanical properties of NR.

All the polymers used have its advantages and disadvantages therefore inorder to obtain the optimum service properties polymer blends were also tried. ENR -50 in blend with SBR and BR are also evaluated for its mechanical properties and abrasion resistance.

Various typical properties of commercial elastomers used in the present work are given in Table 2.[19]

2.8.2 CURATIVES

Vulcanization is a process by which rubber is converted into a three-dimensional network by tying together independent chain molecules. The resulting cross-links can have a monosulphide, disulphide, or a polysulphide structure or both types, depending on the curing system used. Mono and disulphide crosslinks have a relatively high degree of chemical and thermal stability.

However, polysulphide crosslinks are reactive and undergo chemical changes during ageing resulting in changes in physical properties of vulcanizate(product after vulcanization). The most important vulcanizing or curing agent used is sulphur. Vulcanization with sulphur alone is an extremely slow process, relatively large amount of sulphur and large vulcanization times are necessary and the vulcanizate produced are not of high quality. Vulcanization with sulphur alone is therefore of no technical importance at all. The sulphur exists as S_8 ring (rhombic) and is relatively stable. To make sulphur reactive a considerable amount of activating energy must be expended and sulphur ring must be split. The process of activation occurs at high temperature and can be promoted by certain organic substances called accelerators and metallic oxide.

In the present studies rubber grade sulphur along with sulfenamide accelerators 2, 4 morpholine mercaptobenzothiazole (MOR) is used. MOR is fast, safe processing, delayed action accelerators for NR and synthetic rubbers. It is also used to minimize the possibility of sulphur bloom and to obtain vulcanizate having good mechanical properties.

In the case of ENR-50 all the three vulcanization systems were studied. In the conventional sulphur vulcanization system the sulphur dosage is high while the accelerator dosage is low, here in vulcanizates the combined sulphur exists predominantly as polysulphide cross-link. Low sulphur high acceleator system are often called efficient vulcanization (EV) systems because the term 'efficient' derives from the more efficient use of sulphur, i.e., since the crosslinks are shorter and the main chain modification fewer, less sulphur is required to get an effective numbers of crosslinks. It is generally accepted that the EV system at optimum cure can produce a network containing predominantly thermally stable monosulphide crosslinks and vulcanizing is complete and no sulphur is available for further crosslinking. Semi-efficient vulcanization (semi-EV) system represents a compromise between the conventional and EV system.

Activators are materials added to an accelerated vulcanization system to realize in full potential of crosslinks. Zinc oxide and stearic acid are used as the activator system. A higher dosage of zinc oxide is used for importing better heat resistance.

2.8.3 AIDS TO PROCESSING

These are compounding materials that improvethe processability of a polymeric compound. Naphthenic oil and dioctyl phthalate (DOP) are used as plasticiser and softener for proper incorporation of ingredients. Naphthenic oil in non- staining plastcizer, softener especially recommended where colour retention before and after ageing is important. DOP was used as plasti zer and softener for NBR.

2.8.4 AIDS TO QUALITY

A. FILLER REINFORCING TYPE

In general carbon black is used as the reinforcing filler for improving variety of vulcanizate properties. The switch over from carbon black to a less reinforcing filler, silica, was dictated by consumer performance to get as white a rice as possible and introduction of stricter food and drug regulations. Silica offers the highest reinforcement of the nonblack fillers. They are noted for their unique combination of tear strength, adhesion, abrasion resistance, age resistance and colour.

B. ANTIOXIDANTS

Antioxidants are used universally to impart improved and satisfactory ageing properties in order to obtain reasonable service life in the cured compound. The two most important types of antioxidants available are those based on amines and phenolics. The former types are generally more active, but upon ageing they stain or discolour the rubber. The later are less staining but are also less effective for heat ageing and do not confer protection against ozone attack. In the present work amine based antioxidant polymerized 1, 2 dihydro 2,2,4 trimethtylquinoline (A/o HSL) and phenolic based antioxidant styrenated phenol (A/o SP) are used.

2.8.5 SPECIAL MATERIALS

To impart better processability and high hardness coumarone- indene resin (CI resin) is used. To get better interaction between silica filler and the polymer coupling agent bis (3- methoxysilylpropyl) tetrasulfide (SI -69) is used. Since silica adsorbs accelerators and hence retard cure, a special additive like diethylene glycol (DEG) is included in the compound. N- cyclohexylthio phthalimide (PVI) a prevulcanization inhibitor is used to obtain better processing safety with minimal changes in curing characteristics or vulcanizate properties.

For ENR compounding bases such as calcium stearate and sodium carbonate are used to keep the ENR basic and hence to minimize acid promoted ageing.

CHAPTER - 3

EQUIPMENTS AND EXPERIMENTAL PROCEDURE

CHAPTER - 3

EQUIPMENTS AND EXPERIMENTAL PROCEDURE

3.1 EQUIPMENTS

Equipments used for the present work are discussed briefly as below.

3.1.1 TWO ROLL OPEN MIXING MILL

The two roll mixing mill perform both the operations of mastication and mixing. It consists of two rolls revolving at different pheripheral speeds and in opposite directions. The back roll is driven from the gear box or back shaft and the front roll from the back roll through roll and gears. The rolls are made of chilled cast steel, cored to allow for circulation of water for efficient cooling. Another important feature of the mill is the friction ratio i.e., the relative surface speed at which the two rolls revolve. The ratio depends upon the size of mixing mill. Mills are usually fitted with various safety devices, some to protect operators and other to protect the mill.

In the present work, laboratory model (30cm x 15cm) two roll mixing mill from David Bridge and Co., England having a friction ratio 1.25:1 was used.

The two roll mixing mill used in the present work is shown in Photograph 1.

3.1.2 INTERNAL MIXER

The limitations of the two roll mill, pressure on increased production had led to the development of internal mixer. An internal mixer consists of two rollers in the form of interrupted spiral which rotates inside a chamber. The rotors run at even speed and the nogs are designed to produce a friction ratio between the rotors. The work in this machine is done between the rotors. This machine is fitted with a pneumatically operated ram to ensure that the rubber and powders are in contact.

An internal mixer masticates the rubber and mixes the stock rapidly, thus resulting in shorter mixing cycles calling for automation in charging and discharging the batches. It is safer and clean to work with. In the present work laboratory model intermix (Shaw Intermix -MK3, size K0) of capacity 1Lt. from Francis Shaw and Co. (Manchestral) Ltd., England was used.

The diagrammatic sectional view of internal mixer is shown in Figure 5.[15]

3.1.3 THE PRESS

Hand presses and hydraulic presses can be used for the purpose of vulcanization. Two types of presses are used, screw and hydraulic press, screw presses are suitable only for small scale production, hydraulic presses are used for higher production rate and easy operation.

Hydraulic presses vary greatly in size and capacity, depending on moulding requirements. Press platens are heated either electrically or by steam. Electrically heated platens are advantageous when the processesare so few in numbers that maintenance of a boiler is not economical. The location of the elements in electrically heated type should be such as to obtain most uniform heat distribution possible.

In the present work hydraulic press with electrically heated platens of size (18" x 18") from Ind-Expell (India) was used.

The diagrammatic front view of the hydraulic press used is shown in Figure 6.[16]

3.1.4 MOULDS

Moulding is the operation of shaping and vulcanization of a suitable rubber compound using heat and pressure, in a mould which has the desired contours. Its operation includes preheating the mould initially to curing temperature, application of the mould release agent, loading, pressing, curing and stripping. The method, as which the compounded material is to be presented to the mould generally defines the process as compression, transfer and injection moulding.

In the present work the simplest type of compression mould used was the straight a flash type. It is loaded by placing an excess of the uncured stock in the cavity. As the lid is closed under pressure, the compound conforms to the shape of the cavity and after it is filled, the excess material is forced into overflow channels. This type of mould is least expensive and best used in the moulding of relatively simple shapes and has the advantage of better heat transfer than other types.

In the present work moulds made of ordinary mild steel, with specifications as for the relevant test specimen were used.

The diagrammatic view of compression moulding used for the present work is shown in Figure 6.[16]

3.2 PROCEDURE

The various materials used in compounds, their make and suppliers are given in Table 1.

3.2.1 MIXING

The compounds were prepared in two stages. Initial mixing of polymers, fillers and other additives excluding curatives was done in a laboratory model intermix (KO Intermix). Final mixing of curatives was also done separately in the intermix. After final mixing the stock was sheeted out in a laboratory model (30 cm x 15cm) two roll mixing mill.

STAGE - I - INITIAL MIXING (MASTER BATCH)

Masterbatch is a homogeneous mixture of rubber and one or more materials in a known proportions for use as a raw material in the preparation of the final compounds.

Intermix (Shaw Intermix-MK3, SizeK0) was set at rotor speed of 60rpm and an initial chamber temperature of 60°C and ram pressure 3 Kgcm^{-2}, the dump temperature was maintained at 125° - 135°C. The following mixing cycle was used.

TIME (MINUTES)	OPERATION (ADDITION)
0	Polymer (mastication)
2*	Zinc oxide + stearic acid + A/ox HSL + PVI + base (for ENR)
2½	½ filler + A/ox SP + DEG + SI-69 + Cl-resin.
3½	½ filler + Naphthenic oil / DOP.
7	Dump.

* In case of NBR Sulphur was added along with zinc oxide + stearic acid + A/ox HSL + PVI at 2 minutes.

STAGE II FINAL MIXING

Master batches were matured over night and the curatives were incorporated in the internal mixer, set at a rotor speed of 60rpm, initial temperature 50°C and ram pressure 3KG cm^{-2}. It was mixed for 3 minutes and dump temperature was maintained below 100°C. These were then sheeted out in a laboratory model (30cm x 15cm) two roll mixing mill.

In the case of blends the viscosity of ENR was first reduced by mastication on a two roll mixing mill and was then blended with the second polymer at the same viscosity for 2 minutes.

The formulation for mixes for paddy dehusking rubber rollers used in the present work are given in Table 3. (Formulations 1 - 11)

3.2.2 MOULDING OF SHEET

Moulding was carried out using compression moulding in a 18"x18" hydraulic press having electrically heated platens maintained at 150°C. The mould was completely filled with excess compound than the mould volume, closed and then placed in the press. Sufficient hydraulic pressure was applied and cured for 90% of optimum cure time (t_{90}) as obtained from a rheometer R- 100. Test samples were prepared as per relevant standards for testing.

The rheometric analysis for Formulations 1-11 are tabulated in Table 4.

CHAPTER - 4

TESTING

CHAPTER 4

TESTING

4.1 RHEOMETRIC ANALYSIS

The Rheometric R-100 provides a continuous smooth curve of elastic modulus versus curve time. The smooth curve is ideally suited for direct comparative testing.

A polymer compound specimen is contained within the vulcanization chamber under conditions of preset temperature and pressure. The disc is oscillated through a small arc of 3^o exerting a shear strain on the test specimen. The force (torque) required to oscillate the disc is proportional to the stiffness (shear modulus) of the polymers. The stiffness of the specimen increases when crosslinks are formed during curve. A complete curve is obtained when the recorded torque value either increases to an equilibrium value or a maximum value. The time required to obtain a cure curve is a function of the test temperature and the vulcanization characteristics of the polymer specimen.

In the present work optimum cure times at 150°C were determined using a Monsanto Rheometer R-100. The optimum cure time corresponds to the time to achieve 90% (t_{90}) of the curve calculated from the formula.

Optimum cure $(t_{90}) = 0.9\,(L_f - L_i) + L_i$

Where L_f and L_i are maximum and minimum torques respectively. Time to incipient cure scorch time (t_{s2}), is the time taken for the torque to rise two units above the minimum torque. Characteristic curve for rheometric analysis is shown in Figure 7.

The reheometer used in the present work is shown in Photograph 2.

4.2 VULCANIZATE PROPERTIES TESTS

4.2.1 MODULUS, TENSILE STRENGTH AND ELONGATION AT BREAK

These three parameters were determined according to ASTM D- 412-80 test method, using dumb-bell shaped test pieces. The test pieces were punched out from the moulded sheets using D-type die, along the mill grain direction of the vulcanized sheets. The thickness of the narrow portion of specimen was measured using a dial gauge. The specimens were tested in INSTRON-4301 universal testing machine (UTM) at 25±2°C and at a cross-head speed of 500mm per minute. The elongation at break, modulus and tensile strength were recorded.

The UTM used for for the above test is shown in Photograph 3.

4.2.2 TEAR RESISTANCE

The tear resistance of the sample was tested as per ASTM-D 624-81 test method, using unnicked 90°C angle test specimens which were punched out from the moulded sheets, along the mill grain direction. This test was also carried in INSTRON-4301 (UTM), at a cross-head speed of 500mm per minute and at 25±2°C.

The UTM used for the above test is shown in Photograph 3.

4.2.3 HARDNESS

The hardness of the samples was measured as per ASTM D-2240- 81 test method using a Shore A type durometer, which employed a calibratedspring to provide the indenting force. Since the hardness reading decreased with time after firm contact between the intender and the sample, the readings were taken immediately after the establishment of firm contact.

4.2.4 ABRASION RESISTANCE

The abrasion resistance of the samples was tested using a DIN abrader. It consist of a drum on to which a standard abrasive cloth is fixed. The drum is rotated at a speed of 40±1rpm and total abrasion length in 42 meter. Sample having a diameter of 16±0.2mm and a thickness of 6 to 10mm is kept on rotating sample holders. Usually 10N load is applied. Initially a pre-run was given for the sample and then its weight taken. The weight after the final run was also noted. The difference in weight is the abrasion loss. It is expressed as the volume of the test piece getting abraded away by its travel through 42 meter on a standard abrasive surface. The abrasion loss was calculated as follows

$$V = \frac{\Delta m}{\rho}$$

where,

Δm = mass loss, gm.

ρ = density, gm/mm^3

V = Abrasion loss, mm^3

The equipment used for the above test is shown in Photograph 4.

4.2.5 HEAT BUILD UP AND PERMANENT SET

The Goodrich flexometer confirming to ASTM designation ASTM D-623-67 method A was used for measuring heat build-up. The test was used for measuring heat build-up. The test was carried out with the cylindrical sample of 2.5cm in height and 1.9cm in diameter. The oven temperature was kept constant at 50°C The stroke was adjusted to 4.45mm and the load to 10.9 Kg. The sample was preconditioned to the set temperature for 20 minutes. The heat developed was sensed by a thermocouple and relayed to a digital temperature indicators. The temperature rise (Δ T°C) at the end of 25 minutes was taken as heat build up.

The sample was then taken out and the thickness was measured after half an hour. Permanent set was calculated from the residual height of the sample and expressed as

$$\text{Permanent set (\%)} = \frac{t_0 - t_i}{t_0} \times 100$$

where, t_0 and t_i are theinitial and final heights respectively of the test specimen.

The line diagram of the equipment used for the above test is shown in Figure 8.

4.3 ACCELERATED AGEING TESTS

The natural deterioration of the vulcanizate under the action of heat, light, oxygen, ozone etc., is termed as ageing. The service life of a product is too large to wait for getting information regarding the performance of the product under the influence of the above mentioned agents. It is therefore necessary to test the product, under conditions which can produce accelerated ageing effects, to get some idea of the service life and performance of the product.

In the present work ageing test was conducted by keeping the samples in an oven. The samples were kept for 96hr. at 100°C ±2°C and then tested for properties like hardness, tensile strength, tear resistance, elongation at break and modulus as described earlier for original vulcanizate properties test.

4.4 OIL RESISTANCE TEST

(Service fluid)

Oil resistance tests were carried out with service fluid i.e., rice bran oil for comparative evaluation of theeffectof service fluid on the different compounds. The apparatus used for the test is shown in Figure 9.

The samples for different tests were placed in a test tube of outer diameter 45mm and overall length of 300mm filled with service fluid and then closed with aluminum foil. The tube then was kept in oven maintained at $70^{\circ}C \pm 2^{\circ}C$ for 70hr.

The following tests were conducted according to ASTM D471 - 79 test method.

CHANGE IN MASS

$$\text{Change in mass (\%)} = \frac{M_2 - M_1}{M_1} \times 100$$

where M_1 = initial mass of specimen

M_2 = final mass of specimen after immersion

CHANGE IN VOLUME (Dimensional - change method)

Measure the test specimen (2cm x 2cm) for its original length, width and thickness of each test piece denoted as Lo, Wo, To, respectively. After immersion period, remeasure the length, width and thickness denoted as L, W, and T respectively.

$$\text{Change in volume (\%)} = \frac{F\,V - I\,V}{I\,V} \times 100$$

F V = Final Volume = (L W) * T

I V = Initial Volume = (Lo Wo) * To

* – Values measured using graph paper

CHAPTER - 5

RESULTS AND DISCUSSIONS

CHAPTER - 5

RESULTS AND DISCUSSION

5.1 RESULTS

Laboratory studies

Eleven compounds were selected for evaluation and mixing procedure adopte given in Section3.3 and formulations are tabulated in Table 3. Experiments were carried out in three stages. In first stage formulations based on NR, NBR, ENR were studied (Formulations1-4). In second stage formulations based on ENR, ENR blends with BR, SBR, NBR were studied (Formulations 5-9). In the third stage,Compound 6 was studied for effect of increased dosage of stearic acid and A/o HSL (Formulations 10 and 11)

Optimum cure times were assessed by rheometric analysis correspond to the times to achieve 90% (t_{90}) of the cure. For convenience, Rheometer was set at 150°C and 3° arc and same initial conditions for all Eleven formulations. The characteristic graph obtained from rheometric analysis is given in Figure 10 (Formulations 1-4), Figure 11 (Formulations 5-7), Figure 12 (Formulations 7-9). The rheometric analysis reports are given in Table 3.

Testing of vulcanizate properties was also carried out in three stages after preparation of various compounds using compression moulding. Test samples from each compound sheet were prepared as per ASTM and DIN standards and relevant test methods were employed for testing. The moulded test pieces from each compound are taken and tested, at least three specimens per compound were tested for each property. The values of vulcanizate properties, aged properties, oil resistance properties, obtained are given in Table 5.

Formulation 5 was also tested for change after immersion in oleic acid at 70°C for 70hr. and the results are given in Table 6.

5.2 DISCUSSIONS

- NR and NBR compounds were chosen basically as control compounds. NR was used as it is cost effective and NBR because of its service life and rice bran oil resistance NR, though posses good physical properties is inferior in rice bran oil resistance when compared to NBR compound.
- Formulation based on NBR was found to be better in all respects, except the high heat build up. NBR best suited for rice dehusking rubber rolls, as it showed (Formulation 2)

- Good Physical properties
- Better ageing resistance
- Excellent abrasion resistance, and
- Excellent rice bran oil resistance

* It was found that during mixing ENR compound sticks to the mill rolls, therefore for the final mixing also internal mixer was used. Also, for final sheeting out proper cooling system must be maintained in both the rolls in case of two roll mixer.

* ENR vulcanizate showed higher degree of reinforcement with silica filler without coupling agent, (evident from test results, rheometric analysis) in agreement with the reported values. But on addition of coupling agent heat build up was found to be low, therefore coupling agent was used in rest of the formulation based on ENR (Formulations 3, 4)

* In the case of ENR, efficient vulcanization (EV) system and semi EV system showed improved ageing properties as compared to the conventional vulcanization system (Formulations 5,6)

* ENR blend with BR showed improved abrasion resistance, and was found to be better than all other compounds tried (Formulation 7). ENR blend with NBR and SBR showed little change in properties.

* The resistance of ENR-50 to ASTM oils approaches that of medium acrylonitrile (34%) NBR copolymer and is superior to that of polychloroprene rubber[3,4,5] (CR). With this report in mind it was thought fit that ENR - 50 could be a substitute for NBR and may prove to have better service life than NR which are now being used for rice dehusking rolls. An interesting and peculiar phenomenon was observed after testing test specimen with service fluid i.e., rice bran oil. It was found the ENR based vulcanizates were sensitive towards rice bran oil with high acid value, even though they meet IS- specifications. This is evident from the experiment conducted (Table 5).

* It is reported that acid value of rice bran oil increases during storage and depends in general upon the care exercised in handling the oil bearing material prior to extraction of the oil. Since rice bran oil contains unusually active lipase, high acidity is probably inherent in this oil. A free fatty acid contents as high as 4.6% has been found even in the oil which had been properly extracted from bran freshly removed from the rice.[9,21]

The results obtained after oil resistance test showed that the acid value can be the probable cause for poor performance of ENR in service fluid. This can also be seen from the similar results obtained after testing ENR based vulcanizate in oleic acid (Table 6) since rice bran oil contains 13--20% saturated and 80-85% of unsaturated acids which has about 40-45% oleic acid. The results are also compared with that of test in rice bran oil (Table 6).

- The other reason for poor performance of ENR vulcanizate could be that at high acid concentration and temperature, ring opening of the epoxide group may occur which can adversely affect the properties of ENR.

- Formulations based on ENR tried with higher degree of stearic acid and A/o HSL showed no considerable change in vulcanizate properties (Formulations 10, 11)

- Due to unavailability of fresh rice bran oil, oil resistance test have to be conducted using stored rice bran oil and during two different phases of project acid value of rice bran oil has also changed (reported in Table 5.) As it is reported that freshly extracted rice bran oil contains low fatty acid value,[9,21] further work can also be extended on the effect of acid value of the vegetable oils on the ENR based vulcanizates.

- So far no work has been reported on the use of ENR for its resistance towards vegetable oil, further it was found that ENR is sensitive towards rice bran oil with higher acid value. Hence this study has opened up a new field for research on effect of vegetable oil and the acid value on ENR vulcanizate.

TABLES

TABLE 1

FUNCTIONAL CLASS AND SPECIFICATION OF MATERIALS USED

S.N	MATERIAL	FUNCTIONAL CLASS	SPECIFICATION/GRADE	SUPPLIER
1.	Natural Rubber (NR)	Elastomer	ISNR-5	RRII,Kottayam
2.	Nitrile Rubber (NBR)	Elastomer	Chemaperene N-3309	Synthetics & Chemicals ltd.,
3.	Epoxidised Natural Rubber (ENR-50)	Elastomer	ENR-50	RRII, Kottayam
4.	Polybutadine Rubber (BR)	Elastomer	Cisamer-1220	IPCL
5.	Styrene Butadiene Rubbuer (SBR)	Elastomer	Synaperene-1502	Synthetics & Chemicals ltd.,
6.	Silica	Reinforcing filler	INSIL-VN3	Vam Organic ltd.,
7.	Zinc oxide	Activator	Rubber grade	Commercial sample
8.	Stearic acid	Activator	Rubber grade	Commercial sample
9.	Napthenic oil	Process oil	Rubber grade	Commercial sample
10.	Dioctyl phthalate (DOP)	Process oil	Rubber grade	Commercial sample
11.	Diethylene glycol (DEG)	Activator	Rubber grade	Commercial sample
12.	Coumarone indene Resin (CI Resin)	Plasticizer, tackifier	Rubber grade	Commercial sample
13.	Antioxidant Styrenated Phenol (A/oSP)	Age resistor	Rubber grade	Commercial sample
14.	Antioxidant Polymerised 1, 2 dihydro 2, 2, 4 trimethylquinoline (A/oHSL)	Age resistor	Rubber grade	Commercial sample
15.	2, 4 morpholinemercatto benzothiazole (MOR)	Accelerator	Rubber grade	Commercial sample
16.	Sulphur	Vulcanizing agent	Rubber grade	Commercial sample
17.	N - Cyclohexylthio phthalimide (PVI)	Prevulcanization inhibitor	Santogard PVI	Mansanto Co.,
18.	Calcium stearate	Base for ENR	Laboratory reagent	CDH
19.	Sodium carbonate	Base for ENR	Laboratory reagent	NICE
20.	Bis-(3-methoxysilylpropyl) tetrasulphide (SI-69)	Coupling agent	SI-69	Degussa A.G, Germany

TABLE 2

TYPICAL PROPERTIES OF COMMERCIAL ELASTOMERS[19]

PROPERTY	NATURAL RUBBER (NR)	STYRENE BUTADIENE RUBBER (SBR)	NITRILE RUBBER (NBR)	POLYBUTADIENE RUBBER (BR)
Tensile strength (psi)	4000	3500	4000	3000
Elongation (%)	700	700	600	700
Modulus (psi, 300-400 % elongation)	2500	2500	1500	-
Dynamic Properties	Excellent	Good	Poor	-
Permanent set	Low	Low	Moderate	-
Tear Resistance	Excellent	Good	Good	Excellent
Abrasion Resistance	Fair	Good	-	-
Weather Resistance	Fair	Fair	Poor	Fair
Ozone Resistance	Poor	Fair	Fair	Poor
Oil Resistance	Poor	Poor	Excellent	Poor

TABLE 3

PADDY DEHUSKING ROLLER FORMULATIONS

INGREDIENTS	FORMULATIONS (PHR)										
	1	2	3	4	5	6	7	8	9	10	11
ISNR-5	100	-	-	-	-	-	-	-	-	-	-
Chemaprene N-3309	-	100	-	-	-	-	-	50	-	-	-
ENR-50	-	-	100	100	100	100	50	50	50	100	100
Cisamer-1220	-	-	-	-	-	-	50	-	-	-	-
Synaprene-1502	-	-	-	-	-	-	-	-	50	-	-
INSIL-VN3	100	100	100	100	100	100	100	100	100	100	100
Zinc oxide	10	10	10	10	10	10	10	10	10	10	10
Stearic acid	2	2	2	2	2	2	2	2	2	6	2
Napthenic oil	5	-	5	5	5	5	5	5	5	5	5
DOP	-	5	-	-	-	-	-	-	-	-	-
DEG	5	5	5	5	5	5	5	5	5	5	5
CI Resin	6	6	6	6	5	5	5	5	5	5	5
A/o SP	1	1	1	1	1	1	1	1	1	1	1
A/o HSL	0.25	0.25	0.5	0.5	1	1	1	1	1	1	3
MOR	2.2	2.2	2.2	2.2	1.5	2.5	1.5	1.5	1.5	2.5	2.5
Sulphur	2.2	2.2	2.2	2.2	1.5	0.3	1.5	1.5	1.5	0.3	0.3
PVI	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Calcium stearate	-	-	5	5	5	-	5	5	5	-	-
Sodium carbonate	-	-	-	-	-	0.5	-	-	-	0.5	0.5
SI-69	5	5	-	5	5	5	5	5	5	5	5

TABLE 4

RHEOMETRIC ANALYSIS VALUES

Rheometric values at 150°C,3°arc	FORMULATIONS								
	1	2	3	4	5	6	7	8	9
Optimum cure (t_{90}), minutes	13	54	25	20	30	55	35	50	35
Scorch time (t_{52}), minutes	5	5	5	2.5	2.5	1.5	2	2	3

TABLE 5

VULCANIZATE PROPERTIES

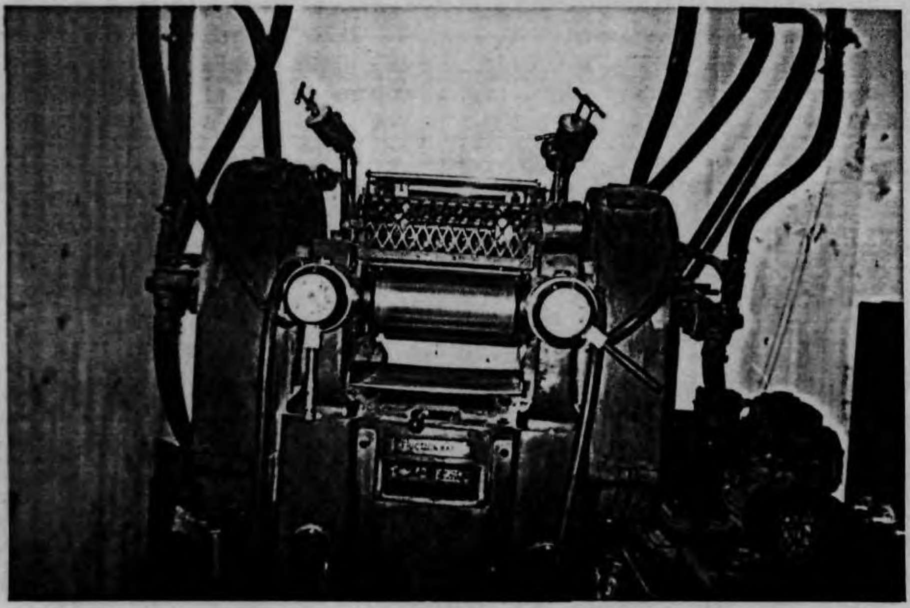
PROPERTY	FORMULATIONS											
	1	2	3	4	5	6	7	8	9	10	11	Is specification
CURED PROPERTIES												
Hardness, Shore A	89	90	92	92	88	90	86	87	85	90	90	85-97IRHD
100 x Modulus (N/mm²)	3.3	4.8	5.4	5.1	4.1	4.4	3.7	6.5	4.7	4	4.3	-
300 x Modulus (N/mm²)	8.2	14.2	13.0	13.0	11.7	12.3	10.1	17.3	14.6	10.2	10.7	-
Tensile strength (N/mm²)	16.5	23.3	21.0	20.0	21.6	15.8	18.8	21.1	18.6	19.4	17.4	8.8 (min)
Elongation, (x)	523	350	373	370	500	386	542	373	366	540	483	130x(min)
Tear strength (N/mm)	80	56	65	71	50	50	88	49	59	45	50	
Abrasion loss (DIN) load lon (mm³)	136	103	184	178	160	145	67	145	132	145	163	-
Goodrich Flexometer heat build up(stroke												
4.45mm, Initial Temp 50°C) (°C)	51	53	51	43	43	50	46	51	47	-	-	
Dynamic Set (x)	9.1	4.2	8.4	5.1	4.3	3.8	5.7	3.7	4.3	-	-	
AGED PROPERTIES, 96hr. at 100°C												
Hardness, Shore A (Points Change)	+3	+3	+4	+3	+2	+4	+4	+4	+3	+3	+4	Max 10 pt
100 x Modulus, (N/mm² Change)	+1.8	+3.52	+10.4	-	-	+8.8	+6	+8	+4.9	+9.2	-	-
300 x Modulus, (N/mm² change)	-	+7.5	-	-	-	-	-	-	-	-	-	-
Tensile strength (x retained)	39	99	80	66	60	95	68	91	88	79	95	-
Elongation (x Retained)	27	90	30	19	22	34	29	37	46	24	19	-
Tear strength (x Retained)	53	97	85	63	79	111	53	84	67	115	114	-
IMMERSION IN RICE BRAN OIL*, 70hr. at 70°C												
Wt. Swell (x)	48	5.1	37	30	115	100	87	55	74	108	116	-
Vol. Swell (x)	60	6.9	40	40	141	116	112	67	83	126	120	-
Hardness, Shore A (Pts. Change)	-21	0	-11	-10	-27	-23	-21	-12	-19	-23	-20	-
100 x Modulus (N/mm² change)	-1.5	+0.2	-2.05	-0.5	-1.85	-1.	-1.5	-1.1	-1.2	-	-	-
300 x Modulus (N/mm² change)	-2.7	+1.6	-	-	-	-	-	-	-	-	-	-
Tensile strength (x Retained)	87	99	32	45	15	26	25	28	24	-	23	-
Elongation (x Retained)	105	104	62	61	13	25	27	35	34	-	19	-
Tear strength (x Retained)	36	98	50	41	7	9	10	20	12	-	13	-

TABLE 6

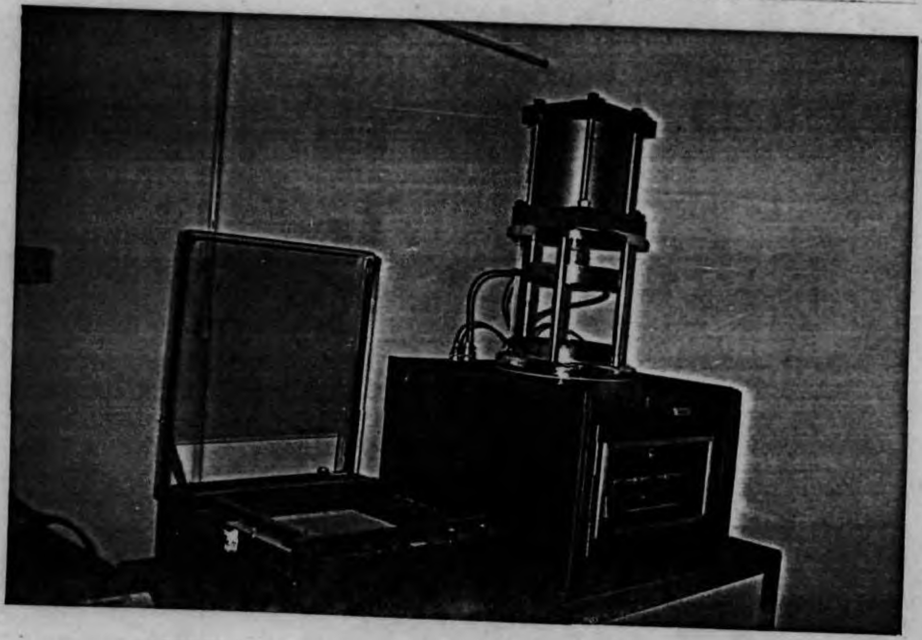
OIL RESISTANCE TEST IN OLEIC ACID

PROPERTY	FORMULATION NO.5	FORMULATION NO.5
	Immersion in Oleic acid at $70^{0}C$ for 70 hr.	Immersion in rice bran oil at $70^{0}C$ for 70 hr.
Weight swell (%)	98.0	115
Volume swell (%)	120	141
Hardness SH A (Pt. change)	-24	-27
Tensile strength (% retained)	16	15
Elongation at break (% retained)	15	13
Tear strength (% retained)	10	7

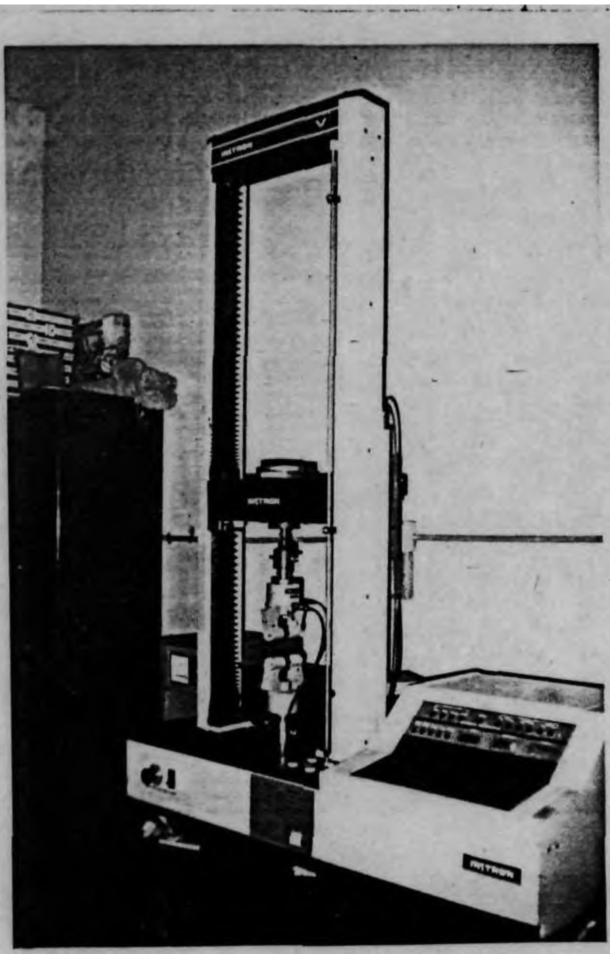
PHOTOGRAPHS



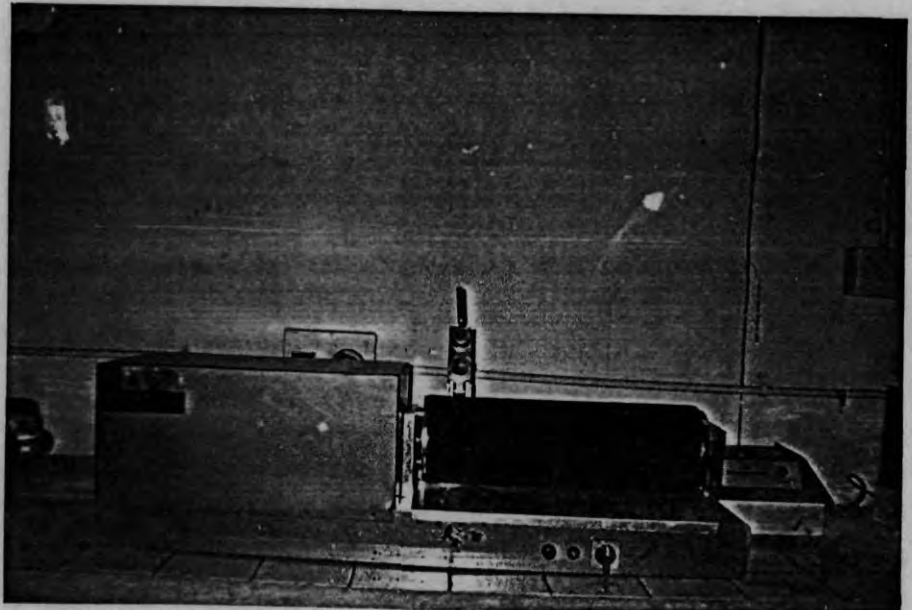
Photograph 1. Two roll mixing mill
(Laboratory model 30cm X15cm)



Photograph 2. Rheometer
(Monsanto R-100)



Photograph 3. Universal testing machine
(INSTRON - 4301)



Photograph 4. DIN Abrader

FIGURES

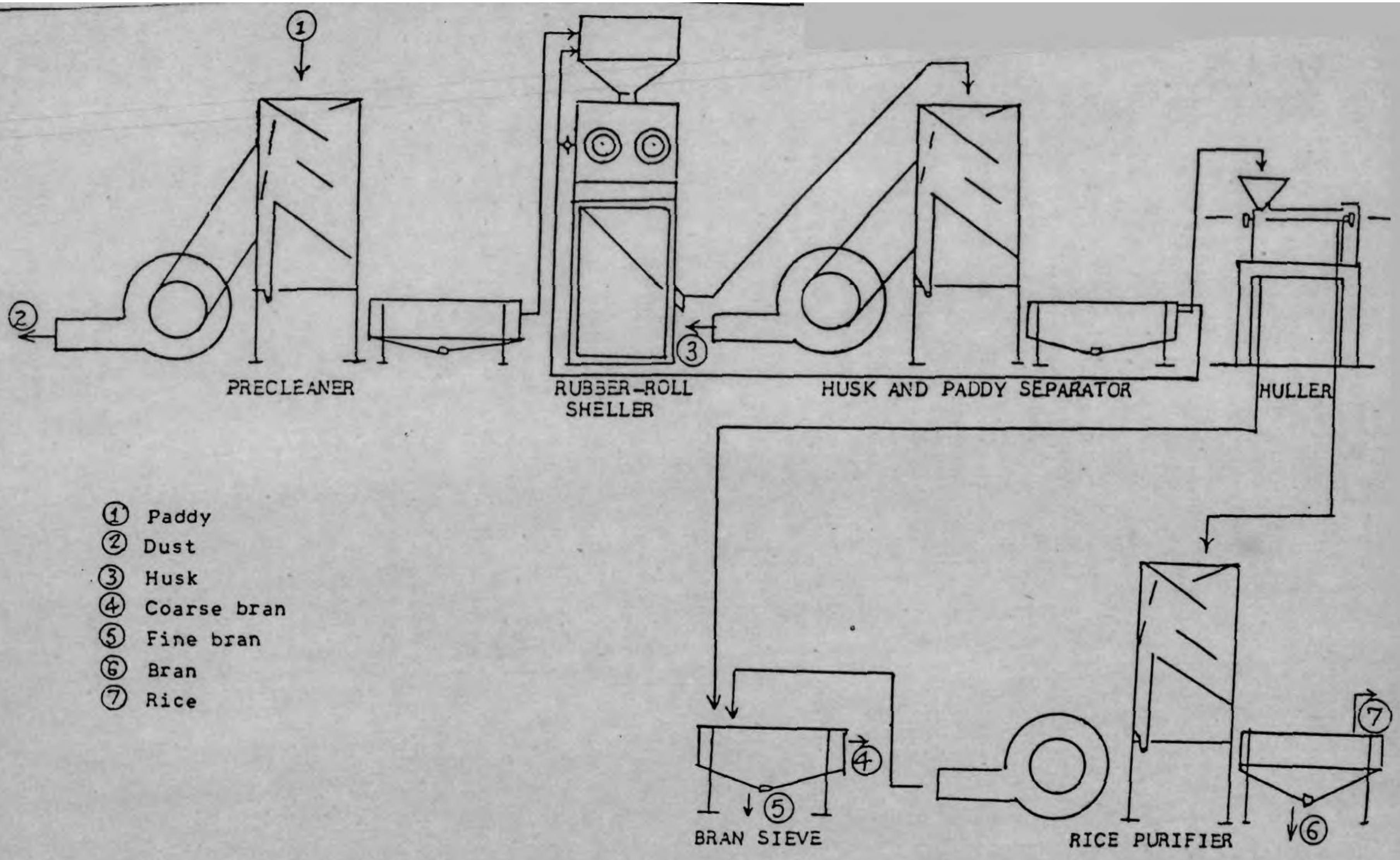


Figure 1. UNITS IN MODERN RICE MILL

Page?

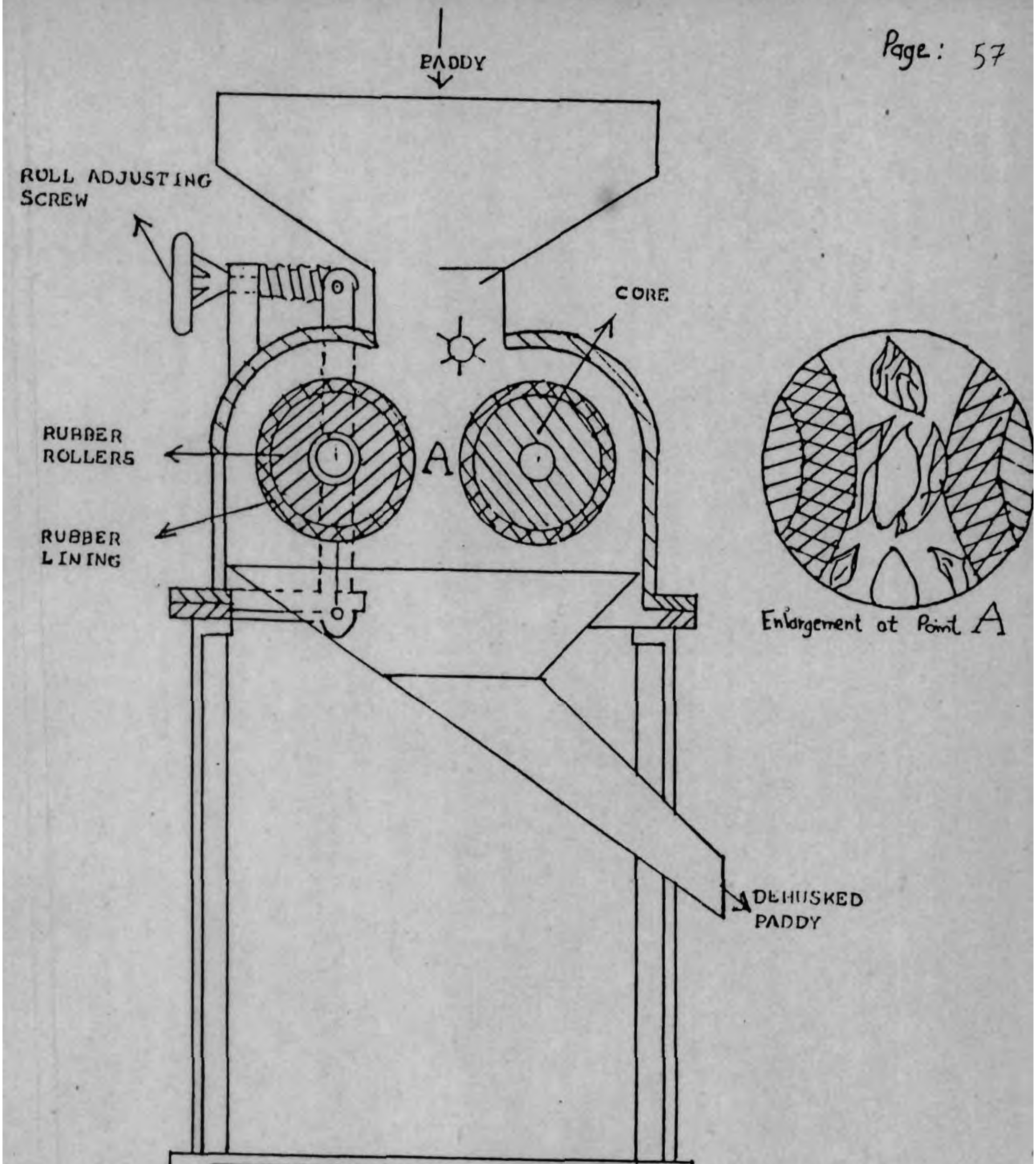


Figure 2. PADDY DEHUSKING

RUBBER ROLLER SHELLER

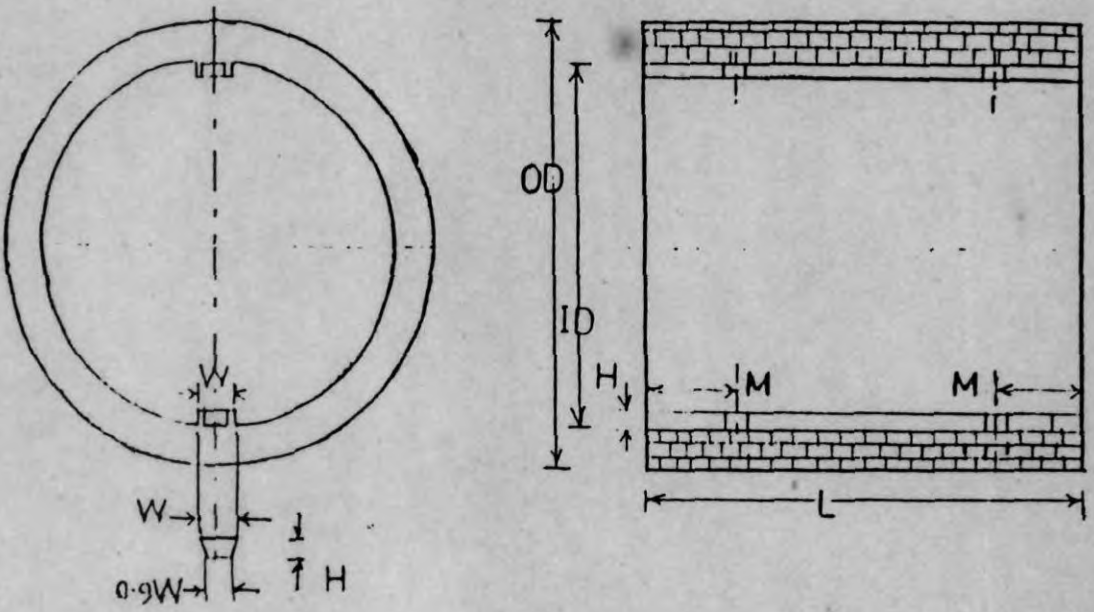


Figure 3. KEY TYPE RUBBER ROLL

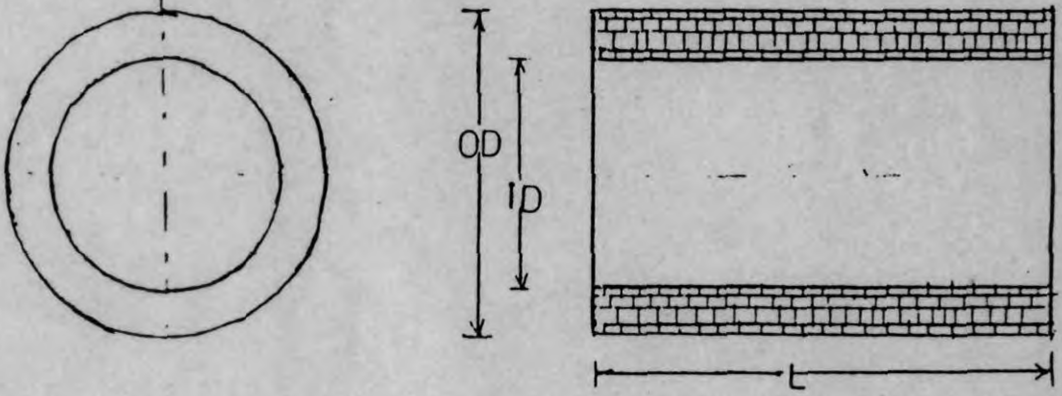


Figure 4. SLIP-ON TYPE RUBBER ROLL

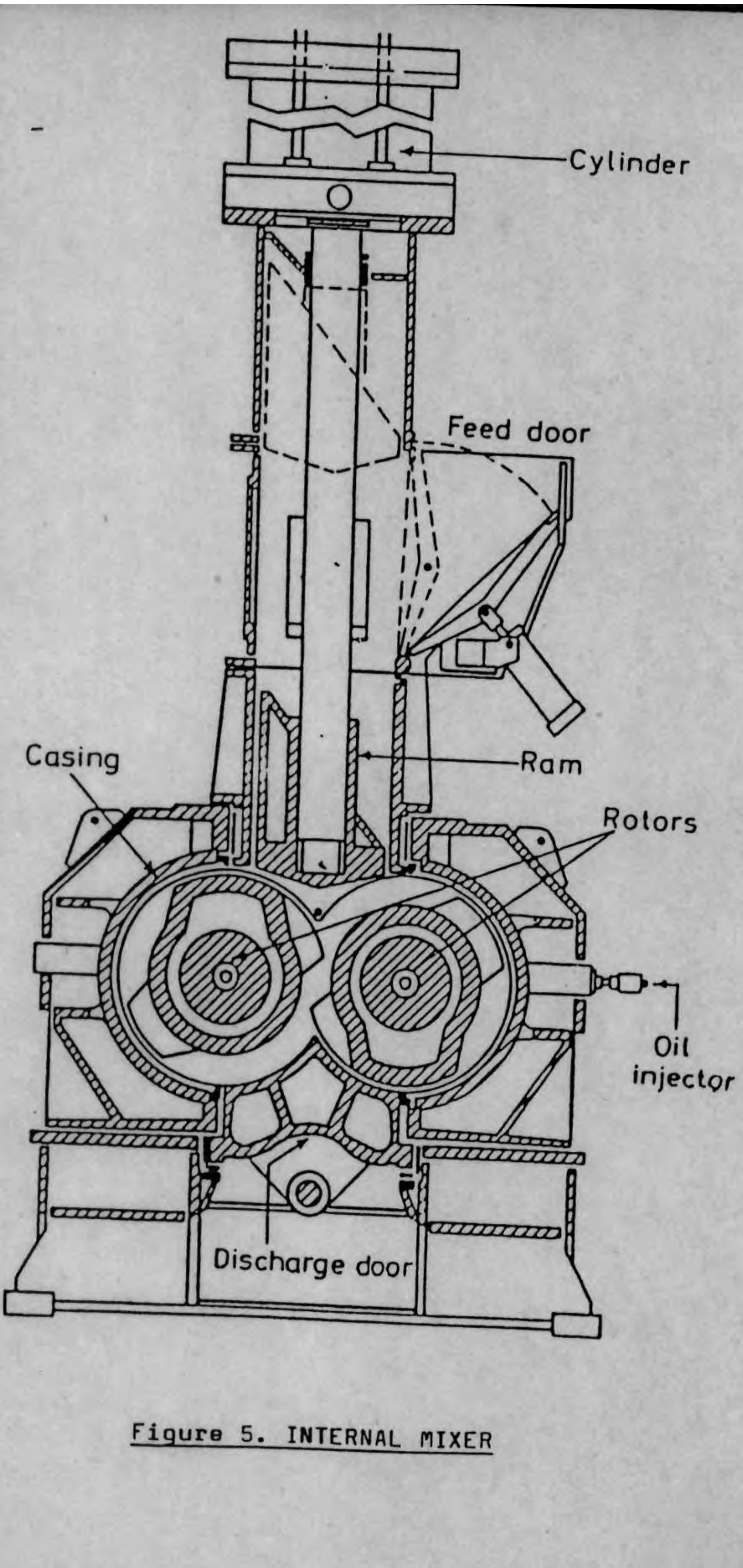


Figure 5. INTERNAL MIXER

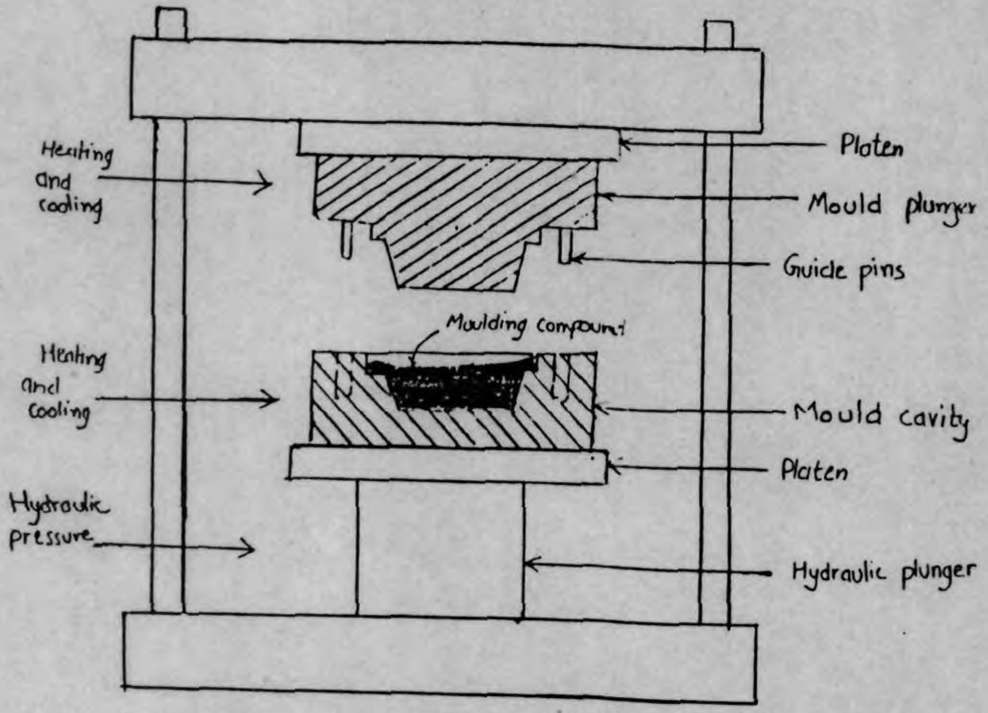


Figure 6. COMPRESSION MOULDING PRESS & MOULD

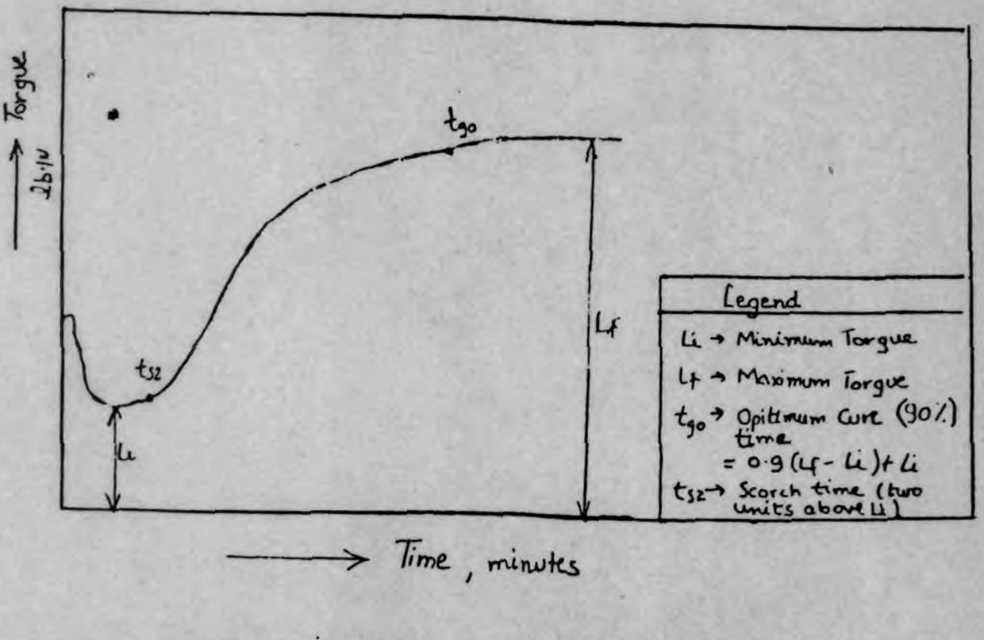
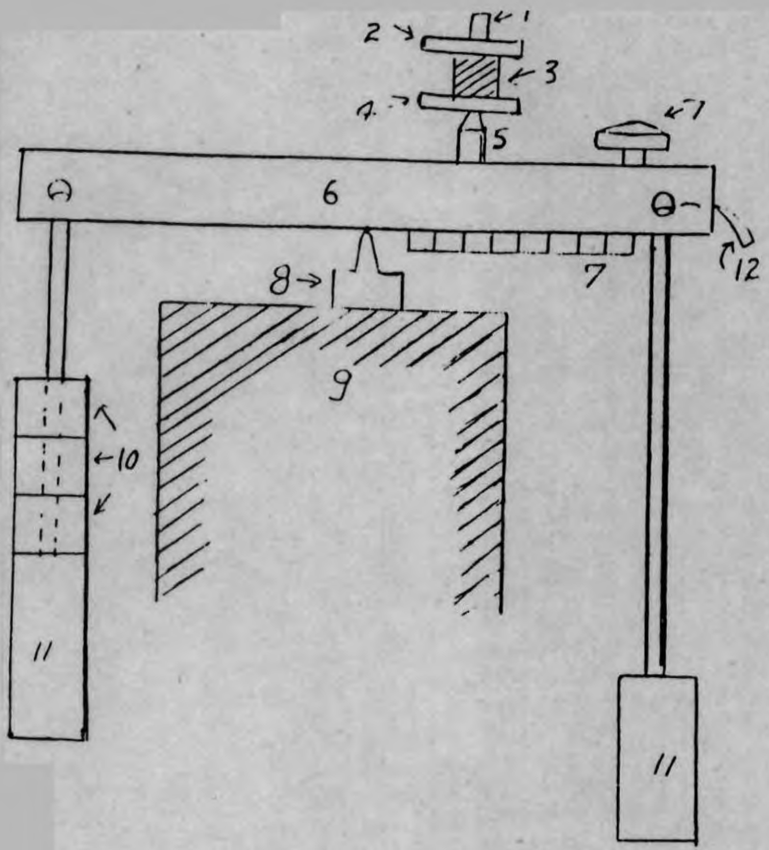


Figure 7. CHARACTERISTIC RHEOMETRIC CURVE



1 - Connection to eccentric which dri
top anvil
2 - Top anvil
3 - Test specimen
4 - Lower anvil
5 - Support for lower anvil
6 - Lever through which load is applied
7 - Calibrated micrometer device
8 - Knife - edge
9 - Supporting base
10 - Test load
11 - Inertia mass
12 - Pointer and reference mark for levelling.

Figure 8. GOODRICH FLEXOMETER

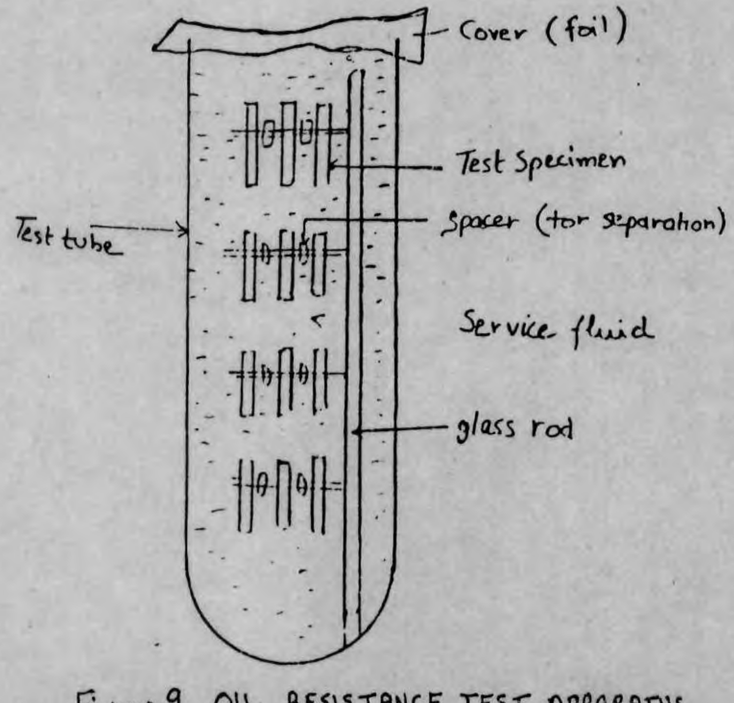


Figure 9. OIL RESISTANCE TEST APPARATUS

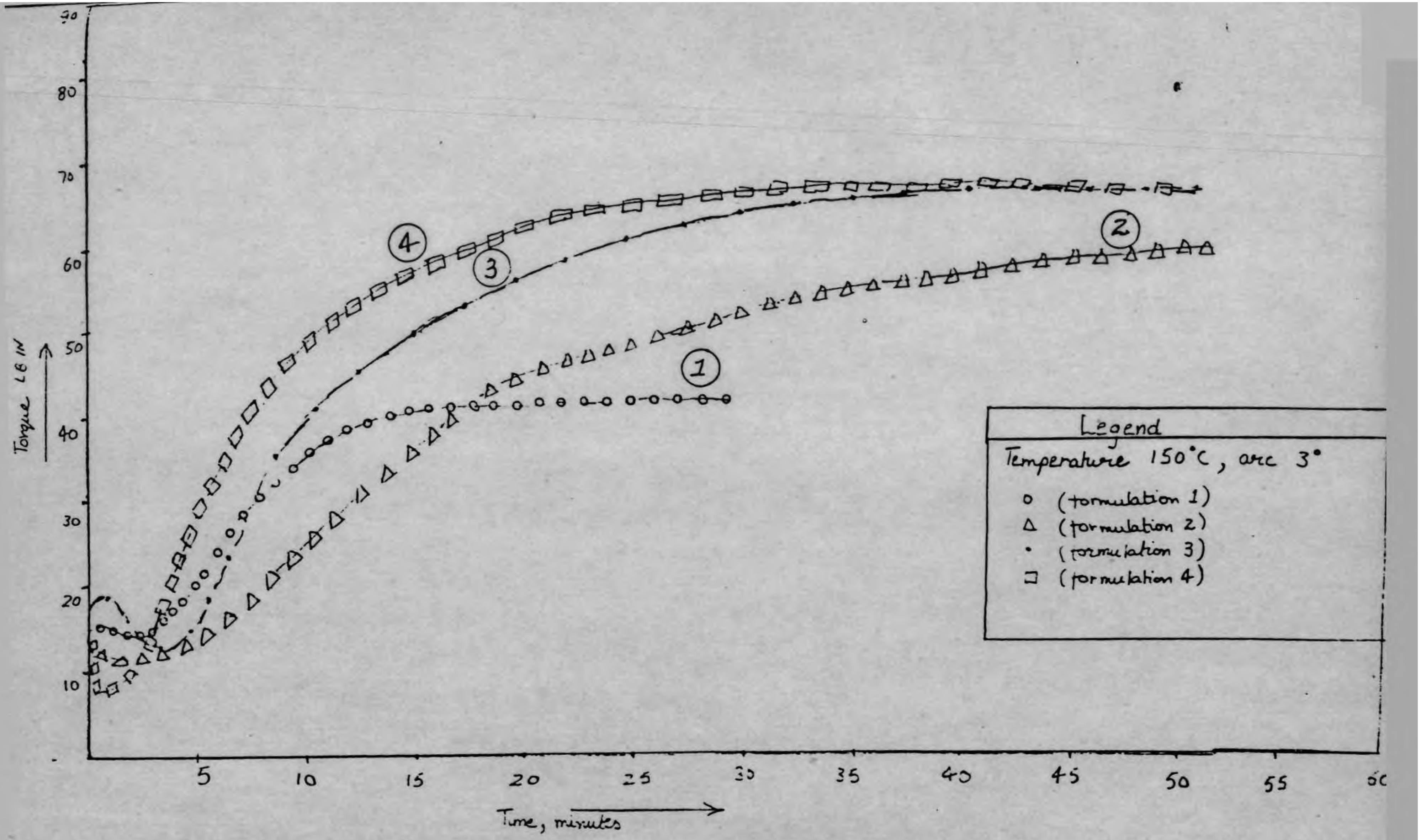


Figure 10. CHARACTERISTIC RHEOMETRIC CURVE (FORMULATIONS 1-4)

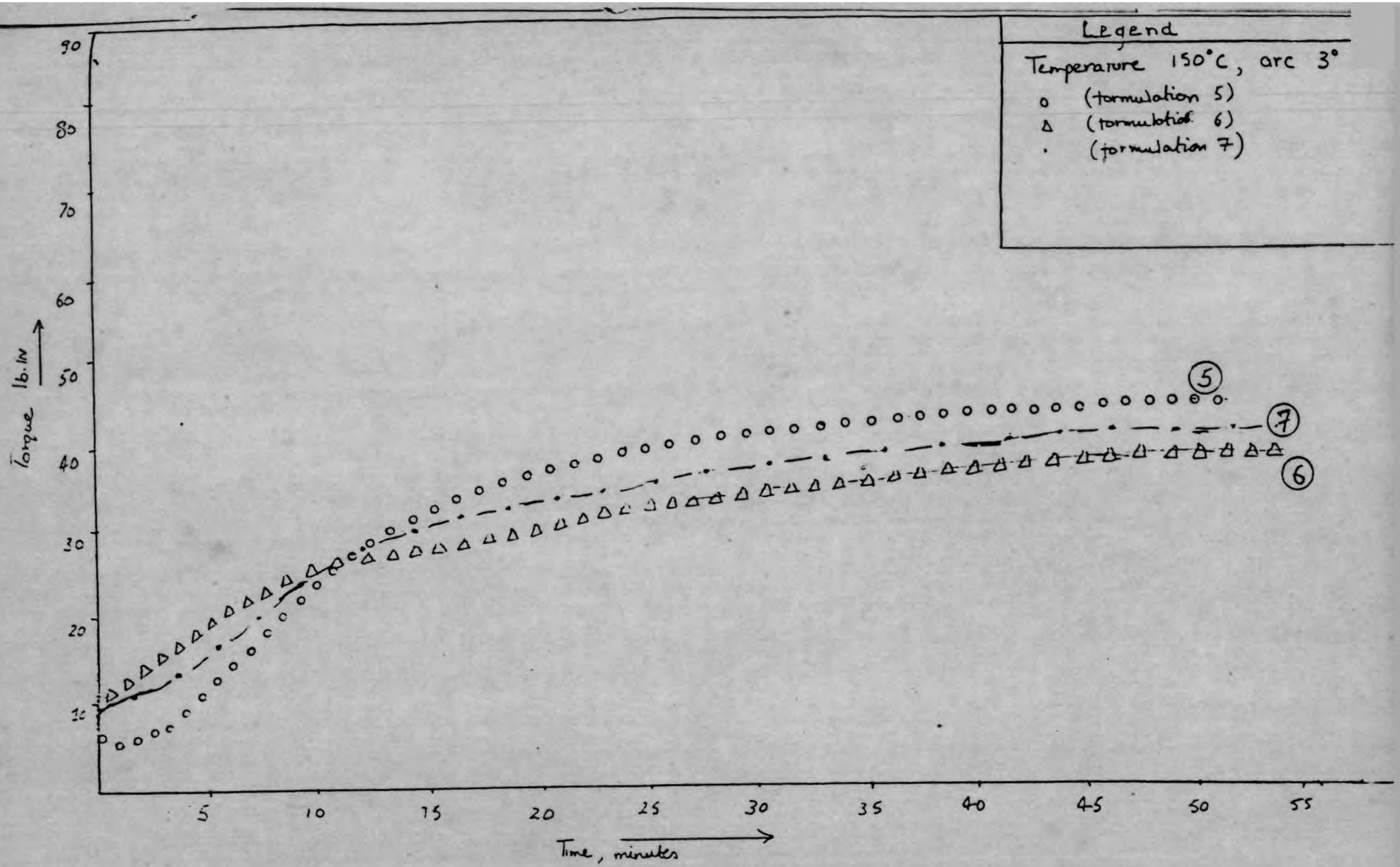


Figure 11. CHARACTERISTIC RHEOMETRIC CURVE (FORMULATIONS 5-7)

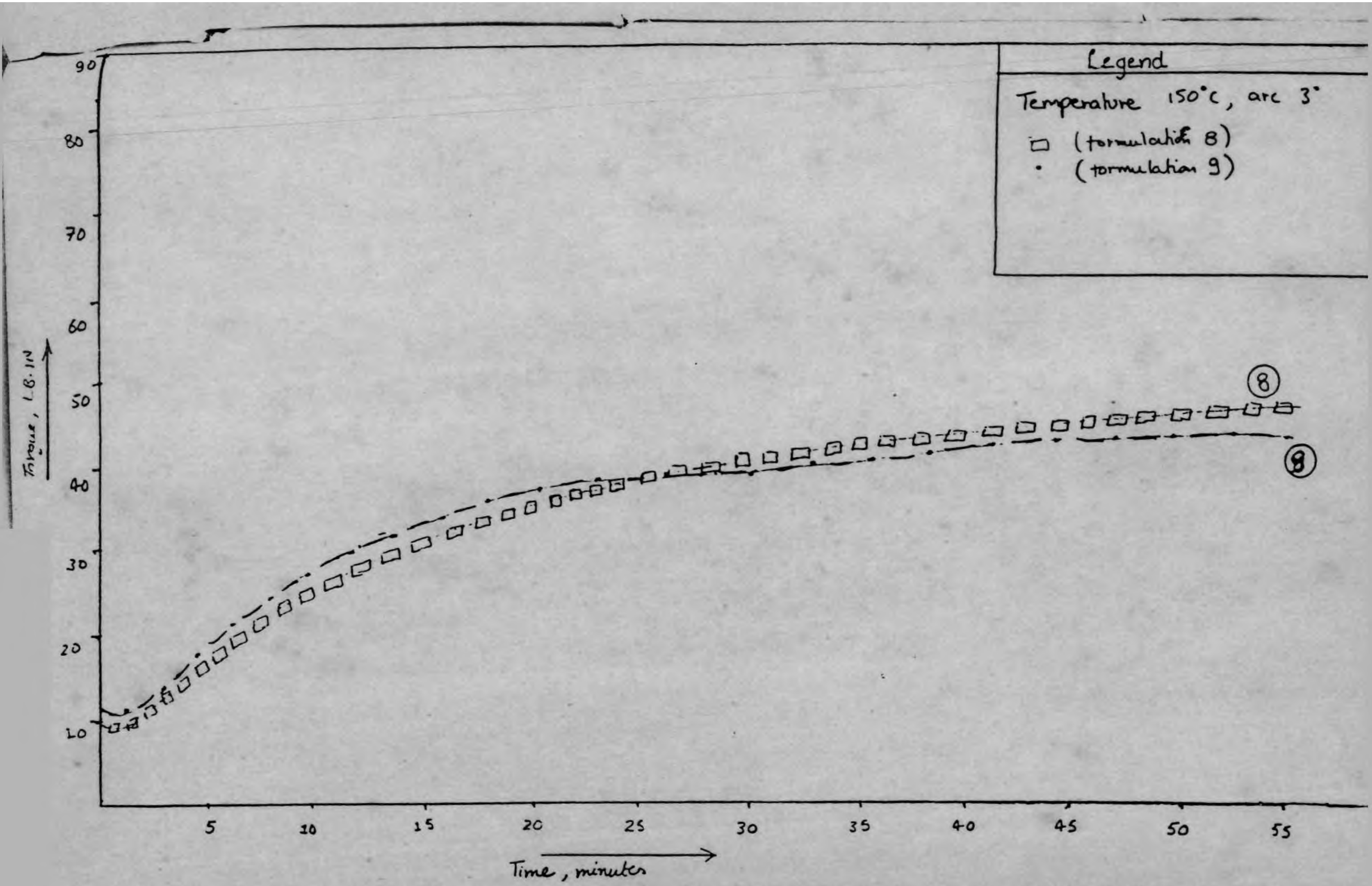


Figure 12. CHARACTERISTIC RHEOMETRIC CURVE (FORMULATIONS 8-9)

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