

# **POLYMER TECHNOLOGY**

**Third Edition**

*by*

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## **PREFACE TO THE THIRD EDITION**

Once again the many advances in polymers and their associated processes have rendered necessary a new edition of this book.

The Material chapters have been updated to include linear low density polyethylene (LLDPE), pearlescent polypropylene, amorphous nylon (Sclar PA), poly (ethylene terephthalate) (PET), ethylene/vinyl alcohol copolymers (EVOH), poly (etherimide) (Ultem), and poly (etheretherketone) (PEEK).

The chapters on Processing have also been updated to take account of developments in powder coating, coextrusion, injection and blow molding, solid phase pressure forming, and thermoforming (including blister and skin packaging). New additions include sections on stretch blow molding, multi-layer (coextrusion) bottle blowing, and the decoration of plastics.

The chapter on Testing has been expanded to include water vapor and gas transmission, while the section on optical tests has been enlarged.

As in previous editions, the applicational aspects have also been updated so that the book will continue to serve the needs of the users of polymers, as well as those of chemists, engineers and technicians in the industry.

## **PREFACE TO THE SECOND EDITION**

Since the first edition of this book there has been a tremendous growth in both polymers and processes.

The framework of the original book seemed to meet with the general approval so this has been retained but a great deal of new materials has been added in order to keep it up-to-date. As well as two new complete chapters on Heat Resistant Thermoplastics and Powder Coating there have been substantial additions to most of the other chapters. Chapter 16, for example, contains information on a number of new materials and has been retitled High Performance Thermoplastics.

Additional materials covered include polyimides, aromatic polyamides, polysulfone, poly (methyl pentene), parylene, polyphenylene oxide and many others. New processes include the molding of structural foams, coextrusion, powder coating, solid phase forming, form/fill/seal bottle blowing, and film fibers.

As in the first edition a great deal of emphasis has been placed on the applications of materials and processes so that the book will continue to be of interest and help to users of plastics and rubbers as well as to chemists, engineers and technicians in the industry.

## PREFACE

The past few years have seen the development of many new polymeric materials, some of them, such as polypropylene and polyisoprene rubber, being the results of completely new polymerization techniques. There have also been corresponding advances in processing techniques. Some of these developments have been the subject of separate textbooks or monographs, while at the other end of the scale there are multi-volume works with each chapter written by specialists.

It seemed to us, however, that there was still room for a book which would cover the most important of these developments in a single volume and incorporate them into the general framework of plastics technology. Originally we were guided to some extent by the syllabus for the plastics technology papers of the Graduateship Examination of the Plastics Institute, since it appeared that there were no single texts available to students. The framework suggested by this approach, namely, Materials, Processes and Testing, remains but with additional material and a slight change of emphasis. These changes should still enable the book to act as a basis for students intending to sit for the Plastics Institute and City and Guilds Examinations as well as the Polymer Technology route to Licenciateship of the Royal Institute of Chemistry.

We have, for instance, included some material on natural, synthetic and modified rubbers following the decision to form a Joint Examining Board for the Plastics Institute and the Institute of the Rubber Industry, at the same time altering the title of the book to *Polymer Technology* from *Plastics Technology*. We also decided to place more emphasis on the applications of plastics, linking these with the properties of the various materials and the conversion processes available. There is an increasing number of people, in a wide variety of industries, who find that they need to know something about the uses of polymers, their advantages and their limitations. Not all of these people have a technical background and we have tried to keep their needs in mind, as well as those of chemists, engineers and technicians.

Finally, for any reader who finds that he needs to know more on any particular topic in this book, we have included suggestions for further reading at the end of each chapter. While these do not include every book written on polymers, they form a fair cross section and can themselves act as stepping stones to more advanced tomes, if desired.

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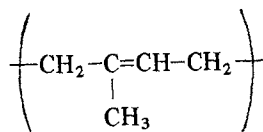
## **PART I GENERAL**



1.

## INTRODUCTION

A polymer may most easily be defined as a material which is composed of comparatively simple recurring units. Well-known examples which occur in nature are the polysaccharides such as starch and cellulose, which are made up of very large numbers of glucose units. Another well-known polymer is natural rubber (polyisoprene); its repeating unit is shown below.



Many other polymers occur in nature but few of them concern us here. As is pointed out in the Preface, our intention in this book is to deal with the technology of certain polymeric materials known as plastics, but for the sake of completeness the subject of rubbers needs to be touched upon.

For a definition of plastics we cannot do better than repeat that given in the British Standards Institute publication, *Glossary of Terms used in the Plastics Industry* (B.S. 1755: 1951) which says, 'Plastics—A wide group of solid composite materials which are largely organic, usually based on synthetic resins or upon modified polymers of natural origin and possessing appreciable mechanical strength. At a suitable stage in their manufacture most plastics can be cast, molded or polymerized directly to shape. Some plastics are rubber-like while some chemically modified forms of rubber are considered to be plastics.'

Rubber is a little harder to define but the following, based on performance characteristics at room temperature, is adequate: 'A rubber is a ma-

terial capable of being stretched to at least double its original length and then returning, rapidly and forcibly, to substantially its original length. An additional requirement is that it shall be capable of being easily formed in its raw state but after vulcanization it shall be able to retain a given size and form.' The theory and practice of vulcanization are discussed in a later chapter dealing with rubbers but it may be briefly noted here that the effect of vulcanization on a rubber is to reduce its plasticity while effectively maintaining its elasticity. Surface tackiness is decreased, while the strength is increased. In many cases it is possible to produce hard materials, as instanced by the material known as 'ebonite.' The first half of the definition above would include many plastics which are, however, excluded because they cannot be vulcanized.

Plastics themselves can be divided into two main subgroups, namely, thermoplastics and thermosets (or thermo-hardening) materials. Thermoplastics are materials which have the property of softening repeatedly on the application of heat and of hardening again when cooled, while thermosets soften once and then harden irreversibly on the application of sufficient heat (usually referred to as 'curing'). When a thermosetting resin is 'cured' it is insoluble in solvents.

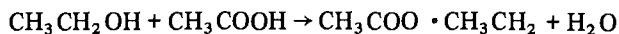
From the definition of rubbers given above, it can be seen that these form an intermediate class of material somewhere between the thermoplastics and the thermosets. Thus, they are thermoplastic during forming operations but are 'cured' or vulcanized after forming to an intermediate elastic or rubbery state and, in some instances, the 'cure' may be prolonged to produce a material closely resembling a thermoset.

The difference in the formation of thermoplastics and thermosetting materials can be explained by reference to the Theory of Functionality, which was first produced by R. H. Kienle. This theory may best be explained using a diagrammatic approach, as follows:

Let the monomer unit be represented as a circle,  $\bigcirc$ , and each functional group (or reactive center) within the monomer be represented by a dash, —.

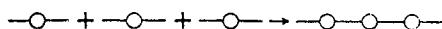
A monofunctional moner ( $f$ ) = 1 would thus be represented as  $\bigcirc—$ .

Since reaction can take place only between reactive groups or centers the only type of compound which can be formed is of the type shown here,  $\bigcirc—\bigcirc$ . An example of this type of reaction would be that between a monohydric alcohol with a monocarboxylic acid to give an ester, e.g.,

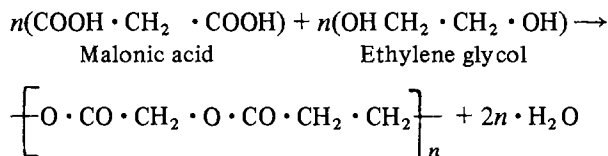


Let us now consider a monomer with functionality ( $f$ ) = 2. Reaction

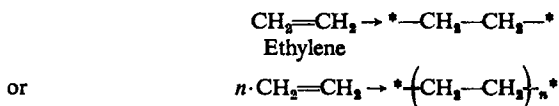
can then continue to give long-chain molecules, in the manner pictured below.



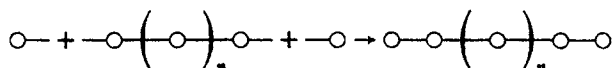
An example of this type of reaction is the formation of a linear polyester from a dihydric alcohol and a dibasic acid, e.g.,



It should be noted, in passing, that a reactive double bond possesses a functionality of 2 since it can open in such a way that it will connect up with two other groups.

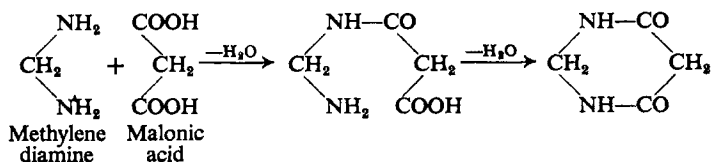


The asterisk denotes that the attached bond can react with other reactive groups which may be present. For a treatment of the kinetics governing such reactions one must refer to textbooks on physical chemistry but it should be emphasized that the reaction is not a straight build-up of one chain only. Very many chains are in the process of formation at any instant and each chain competes for monomer with all other chains. Chain formation ceases when the supply of monomer is exhausted or when reaction occurs with a monofunctional molecule, such as a catalyst residue, solvent molecule or an impurity. Monofunctional compounds are also added deliberately to control the chain length (and hence the molecular weight) of the polymer.



We have said that only molecules of a functionality of 2 or higher can react to form a polymer but while this is true, it does not necessarily mean

that di-functional molecules will always polymerize. Sometimes a functional group is shielded by other groups, and in some cases the formation of a ring compound is favored, either by the geometry and nature of the reacting molecules or by the reaction conditions. If a six-membered ring can be formed, for instance, by intraesterification, then polymerization will rarely occur. Reaction in dilute solution also favors ring formation since the reactant molecules are separated from each other by the solvent molecules, and the chance that the ends of the same molecule will come together is greater than that of their meeting, and reacting, with different molecules. The reaction of methylene diamine and malonic acid, for example, leads to the formation of a six-membered ring as shown below.

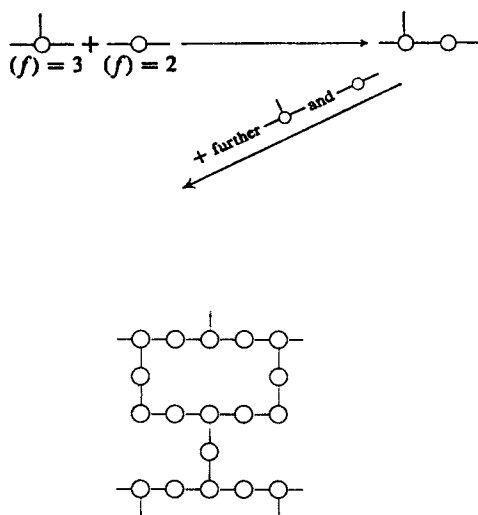


Once the molecule with reactive ends is formed, ring formation is an easy subsequent step. Six-membered rings are the ones most easily obtained because such rings are formed with the least distortion of the bonds connecting the individual atoms. Closer study of these and similar factors governing the formation of polymers is more properly the province of the physical chemist: some excellent texts are available and are given in the reading list at the end of this chapter.

The linear polymers formed by the reaction of difunctional molecules are members of the subgroup of plastics which we have already defined as thermoplastics. The long-chain molecules are held together by relatively weak intermolecular (or Van der Waal) forces, and energy, in the form of heat, will cause the molecules to move relative to each other and the material will then flow. Differences in the softening point of different plastics are due to the relative strength of their intermolecular forces. Polar molecules, such as those of nylon, have greater attraction for each other than nonpolar ones such as polyethylene, and the softening point of the former is, therefore, greater than that of the latter. As is usual in the polymer field this is not the whole story and we find that the softening point

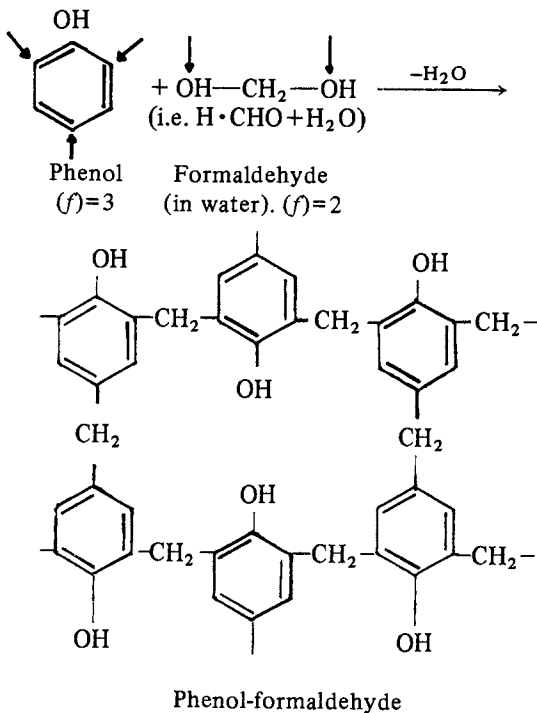
of polystyrene is lower than that of polyethylene in spite of the fact that polystyrene, too, is a polar molecule. This is because there are bulky groups attached to the polystyrene molecule, which prevent a close approach of the polymer chains with a consequent lessening of the attractive forces between them. Less energy (i.e. heat) is necessary, therefore, to cause relative movement or slipping of the chains.

We may now take a brief look at the consequences of including a material with a functionality of 3 among our reactants. Using the pictorial representation referred to previously we see that it is possible to build up a complete network. The diagram shown below is, of course, a two-dimensional representation of what is, in reality, a three-dimensional network.



An example of the type of reaction shown above, where the reactants have functionalities of 3 and 2, is given by the formation of phenol-formaldehyde resins.

The three-dimensional type of polymer typified by phenol-formaldehyde exhibits the type of behavior described earlier as thermosetting, the fully cured compound being unable to flow under the action of heat. Each carbon atom in the network is bound by primary valence bonds to



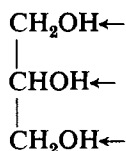
other carbon atoms so that relative movement is almost impossible. In addition, such a structure prevents penetration by solvent molecules and subsequent dispersion so that although solvents may sometimes cause swelling of thermosets they cannot produce a solution.

Intermediate stages are possible in the above and similar reactions, and advantage is taken of this fact in the fabrication of articles from the raw materials. If we consider Stage 1 to be the existence of the starting materials in their monomeric form, and Stage 3 the completely cured compound, then we can visualize a Stage 2 when the full network has not yet been built up and some flow is still possible under the influence of heat. The resin manufacturer stops the reaction at Stage 2 and sells the product in the form of pellets or as a powder. The molder takes this intermediate product and forms it into the required shape, using heat and pressure. The



conditions used to promote the flow in the mold are also sufficient to cause the completion of the fully linked network so that the shape, once formed, is permanent.

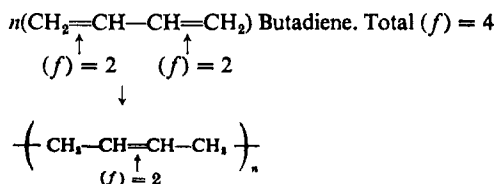
During the discussion on the case of the reaction between two difunctional materials it was mentioned that polymerization would not always take place because a functional group might be inoperational due to the shielding by other groups. The same arguments apply to reaction between di- and tri-functional groups, an excellent example being the reaction between glycerin and phthalic acid. Glycerin is a tri-functional compound, as can be seen from its formula, namely,



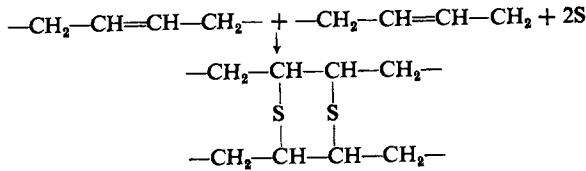
the functional groups being arrowed.

However, the central OH group is, to some extent, shielded by the other two, and temperatures of between 250 and 270°C are necessary to cause reaction of all three OH groups. At lower temperatures (200–240°C) a linear product is obtained, as one would expect from two difunctional compounds.

In passing we may mention compounds having a functionality of 4. A common class of tetrafunctional compound is that of the dienes (compounds having 2 double bonds). The normal reaction of compounds where the 2 double bonds are separated by one single bond (normally referred to as conjugated dienes) is as a difunctional compound but leaving a residual double bond in the molecule.



The residual functionality of 2 can be utilized to combine with material such as sulfur to produce three dimensional networks.



This is the basis of vulcanization of polybutadiene and other rubbers.

The structure of rubbers is intermediate between that of the thermoplastics and thermosets and can best be described as a loosely linked net-

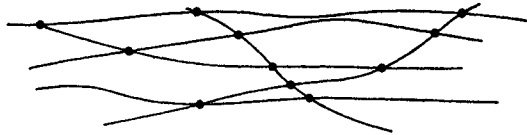


Fig. 1.1

work where the linkages are comparatively few in number (Fig. 1.1).

Such a cross-linked structure is formed by vulcanization of rubbers and allows considerable extension to occur, without breaking the chemical bonds, when submitted to a tensional stress. On releasing the stress, the rubber tends to resume its original configuration.

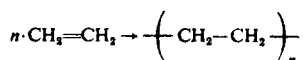
The requirements for fiber formation are, as one would expect, very different and can be summed up as follows: (a) the molecules should be linear and of fairly high molecular weight; (b) the molecules should possess a fairly high degree of orientation; (c) the molecules should have a high degree of linear symmetry, i.e. there should be an absence of bulky side-chains which would prevent orientation and crystal packing; (d) the molecules should contain polar groups.

## POLYMERIZATION REACTIONS

We have already dealt very briefly with polymerization reactions and have classified them on the basis of the properties of the resulting polymers, i.e. thermoplastic and thermosetting. There is, however, a further classification on the basis of the type of chemical reaction which occurs, namely, addition or condensation.

*Addition Polymerization*

In addition polymerization, the resultant polymer is obtained by the combination of a number of simple basic substances (monomers) without loss. Addition polymers are usually thermoplastics. The monomers contain a double bond and, with the exception of fluorinated compounds, always contain the methylene group,  $\text{CH}_2$ . In other words, the basic monomer in addition polymerization is of the general formula  $\text{CH}_2 = \text{C} \cdot \text{R}_1 \text{R}_2$  except, as already stated, in the case of  $\text{CF}_2 = \text{CF}_2$  and  $\text{CF}_2 = \text{C} \cdot \text{ClF}$ . The explanation appears to lie in the relative sizes of the atoms, that of fluorine being the smallest in size, next to hydrogen, which will combine with carbon. Probably the best known example of addition polymerization is that of ethylene to give polyethylene.



There are four basic methods of carrying out addition polymerization and these have certain advantages and disadvantages which are characteristic of the method, irrespective of the monomer being polymerized. In order to facilitate reference to these methods, when considering the formation of the various addition polymers in greater detail later in the book, a brief description and evaluation of each method is given in this chapter.

(a) *Bulk Polymerization.* This is the simplest of the four processes since pure monomer only is used, except for the addition of a catalyst when necessary. The mixture is usually stirred in a large reaction vessel, with or without the addition of heat. The chief advantage of this method is obviously the purity of the final product with the concomitant advantages of excellent color and clarity in transparent polymers such as polystyrene. Bulk polymerization may also be considered to be economically attractive, since the raw material costs consist only of the monomer, but the difficulties of manufacture may sometimes balance this. These manufacturing difficulties arise from the fact that addition polymerization reactions are usually exothermic in nature and with large quantities of undiluted reactants there is, thus, an acute problem of heat dispersal. In addition, as the reaction proceeds, there is an increase in viscosity, and stirring becomes first difficult and eventually impossible, if the reaction is allowed to go to completion.

(b) *Solution Polymerization.* The monomer and catalyst are dissolved in an inert solvent the resultant solution being heated and stirred. In gen-

eral, both heating and stirring problems are eased in solution polymerization, which is also more suitable for continuous processing than is bulk polymerization. There are, however, serious economic problems due to the use of large quantities of expensive solvents which have, therefore, to be recovered and separated. The purity of the resultant polymer is lower than with bulk polymerization because, even with nominally inert solvents, there is frequently some reaction with the active centers of the growing polymer chain, which leads to the existence of solvent fragments in the finished molecule. The polymer is also soluble in the solvent and the removal of the last traces of solvent is rather difficult and expensive. For this reason the solution process is often resorted to for those applications in which the polymer is required in solution form. Surface coatings and impregnating compositions are among the commonest examples where the polymer is applied as a solution, thus rendering evaporation of the original solvent unnecessary.

(c) *Emulsion Polymerization.* In this process the continuous medium is usually water, and the monomer is dispersed instead of dissolved as in the previous process. The resultant polymer is also insoluble in the medium and is thus precipitated as it is formed. The catalysts used are those which are soluble in the dispersing medium, and emulsifying agents such as sodium or potassium salts of fatty acids are added to stabilize the emulsion. After the reaction is completed, the precipitated product is washed in order to remove emulsifying agents, but these are difficult to remove completely and some contamination always remains. There are, of course, few heat transfer problems in such processes because the heat is mainly carried away by the water. One other factor of interest in this process is that it produces the highest molecular weight product.

(d) *Suspension Polymerization.* The monomer in this process is suspended in the carrier medium (usually water) together with a monomer-soluble catalyst. When the mixture is stirred, the monomer is broken into small droplets, each of which will contain a little of the catalyst. A stabilizer, such as methyl cellulose, or starch, is also included in the mixture in order to keep the particles separate from each other during the reaction. These stabilizers are easily removed after the reaction by washing or flotation, leaving the polymer in the form of small beads or 'pearls.' This process can be looked upon as a large number of tiny 'bulk' polymerizations without the disadvantage of heat removal problems. The molecular weight of suspension process polymers is also fairly high.

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