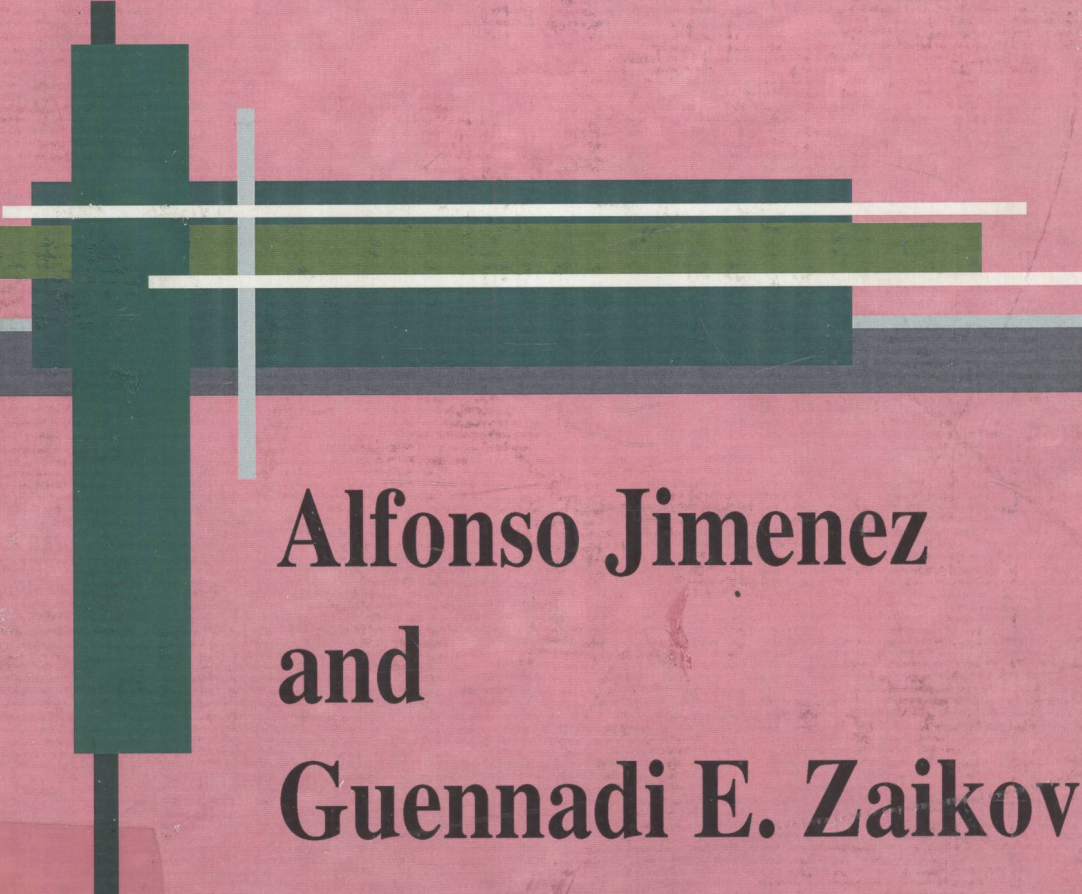


POLYMER ANALYSIS AND DEGRADATION



**Alfonso Jimenez
and
Guennadi E. Zaikov**

NOVA

Science Publishers, Inc.

063 1.1-53
P783.2
1999

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E200100836

Nova Science Publishers, Inc.
Huntington, New York

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Circulation:	Anna Cruz, Ave Maria Gonzalez, Ron Hedges, Andre Tillman

Library of Congress Cataloging-in-Publication Data
Available upon request

ISBN 1-56072-861-2

CIP

Copyright 2000 by Nova Science Publishers, Inc.
227 Main Street, Suite 100
Huntington, New York 11743
Tele. 631-424-6682 Fax 631-424-4666
e-mail: Novascience@earthlink.net
Web Site: <http://www.nexusworld.com/nova>

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Printed in the United States of America

POLYMER ANALYSIS AND DEGRADATION

PREFACE

“Intimate relations are the fine art.
To succeed one must improve his
skills permanently”

Charles Spencer Chaplin¹

Great Chaplin knew what he was talking about. His last marriage brought to him eight children. And Great Man is the great in everything. His remark is valid being addressed also to the problem of Polymer Degradation and Stabilization. Only if understanding of intimate (elementary) reactions in degrading polymer matrix is perfect, it is possible to extend the lifetime (service and storage time) of polymer goods, to increase the production of polymers and to improve their quality. If mechanism of polymer aging is known in details, even more important goal can be achieved. This is the prediction of material behavior and alteration of material properties at service and/or storage conditions. Such prediction is very important for polymers serving for decades.

As quantity of polymers and polymeric products grow, new problem arises, which is polymer destruction and recycling. At this point, the understanding of degradation mechanism is absolutely necessary. The main method of destruction of polymer waste is their incineration producing toxic compounds, harmful for all living. “There are only two choices: or people will reduce the concentration of smoke, or smoke will reduce the population” (G. Batton). Analysis of polymers and polymeric products is absolutely required at all stages of study of degradation, including environmental factors.

The current volume is directed to the analysis of this problem: Degradation and Stabilization. The volume includes papers presented at Conference, which was held in Alicante (Spain) on September 29 - October 1, 1999.

Once former President of the United States said: “Policy is not too bad. If you succeeded, it brings you glory and honor. If you did not, you write a book”.² Hopefully, the Conference in Alicante was important both as a step forward in understanding of fundamental and applied aspects of polymer aging, and as a possibility to write a book.

¹ Probably this epigraph does not repeat words of the Great Actor, but it certainly follows his idea. Whereas Chaplin could make his remark in English, our source was Russian translation of the French version. The case is known how “Notre Dame de Paris” was translated as “Our Lady from the Paris”. We did not make such a mistake.

² Translated from Russian back to English. The meaning has remained although words might not be the same

“I had chosen my way, and nothing would force me to turn aside” (Emmanuel Kant, German Philosopher). Let us go our way, the way of improvement of polymeric materials quality.

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POLYMER ANALYSIS, DEGRADATION AND STABILIZATION

One of the most important features of the technical progress of this century has been deep and wide penetration of polymeric materials into all fields of human life. But, their more than a half century of use has revealed some shortcomings common to all of these materials. Probably the most important of these is the tendency of most synthetic polymers to degradation. Polymer degradation is, in fact, a series of chemical and physical transformations occurring in the polymer during service, processing or storage, which causes deterioration in valuable properties.

Many factors are responsible for polymer degradation, including heat, UV radiation, biological agents and many others. These factors, either separately or together, cause complex processes including cracking, depolymerization, cross-linking, oxidation, etc. They may cause considerable and often quite rapid deterioration of polymer properties leading to a sharp decrease in their useful life.

It should be remembered that a real polymer is a complex system including, apart from the main component (a high-molecular compound), various additives such as plasticizers or stabilizers. Therefore, the investigation of degradation must necessarily cover a wide range of problems in the fields of chemistry and physics of radical, ionic and molecular reactions in a multicomponent system.

At present the world's output of various plastic materials amounts to over 100 million tons annually, and their protection from degradation, i.e. stabilization of polymer properties, is therefore an extremely important economic problem. But, the importance of the problem of polymer stabilization is not solely economic. In many cases a long service life is a technical prerequisite. Further, many polymer products cannot be processed into finished articles, unless previously stabilized.

Processing of polymers and use of a large variety of polymeric products are rapidly developing and increasing, respectively, all over the world. This progress is simultaneously accompanied with broad environmental and health concerns. This means that there is significantly increasing interest in and demand for new and improved polymeric material formulations and processing technologies with less environmental pollution and fewer harms to the health of humans. As a consequence, the focus of research and development efforts from analysis and protection of the environment is increasingly turning towards the direction of investigating potential new environment friendly polymer compositions and processing methods for these products.

The maturation of the field of synthetic polymer chemistry, as well as the development of an improved understanding of structure - property relationships, has resulted in the ability to synthesize materials with properties designed for a particular application. This versatility makes these high temperature - high performance organic polymers attractive for aerospace, microelectronics and other industrial applications. Current uses include films in semiconductor applications, matrix resins in carbon fiber reinforced composites, foams for insulation, ablatives, adhesives for metals and composites, fibers for sporting goods and membranes for industrial gas separation.

An additional problem associated to the degradation of polymers is the incineration of plastic wastes, which is connected with a number of environmental problems. One of these problems is the formation of toxic combustion by-products. Formation of dioxins and chlorinated compounds are reported from poly-(vinyl chloride), nitrogen oxides from polyamides, etc. Incineration of plastics occurs via a complicated integration of thermal degradation, pyrolysis, combustion of pyrolysis products, mass and energy transport. One essential step in the whole process is the thermal degradation or pyrolysis, respectively, which determine the rate of evolution of gaseous products from plastics. The rate of evolution of gaseous products affects the consecutive steps of the incineration process, i.e. in determining the locally required air supply. From this, the knowledge of the kinetics of thermal degradation and pyrolysis of plastics is essential for a proper design and operation of incinerators. This new problem is also considered in this volume.

This volume is organized to provide a wide coverage of new and traditional polymers and their behavior during degradation, as well as the important chemical and engineering aspects of these materials relative to processing and performance in various applications. This volume starts with some introductory lectures designed to give an overview of the entire field. The remainder of the volume is organized into three sections: Properties; Processing and Modeling, and Applications and New Materials. Each section describes leading research and development that encompass a variety of materials, experimental techniques, theories, processes and applications. Work is included from an international group of scientists and researchers.

The breadth of the information given in this volume should make useful for materials scientists, polymer chemists and engineers in the Universities as well as in the aerospace, automotive, chemical and electronics industries. We hope that this volume will become a useful resource for a broad spectrum of scientists and engineers whose work concerns the preparation, processing, properties and applications of polymers and polymer composites.

Some topics for the proposed sections in this book on degradation and stabilization of polymers were selected by the authors in order to cover fundamental and applied subjects in those interesting aspects of polymer science and technology. Those topics were selected by consideration of the general interests for academic and industrial researchers.

This volume is mainly devoted to kinetic studies in polymers, not only chemistry but also biology, medicine and agriculture. It will permit the consideration of another important factor, which is the education in thermal analysis kinetics at training courses and conferences organized by Universities. It seems that there are very few teaching materials available for such experiments on thermoanalytical kinetics. Therefore, it is evident that versatile teaching materials on the kinetics of solid state reactions should be developed to foster the new generation of scientists working in this field. As an answer to such question this volume is the result of the First Workshop on Polymer, Analysis, Degradation and Stabilization, which was held in Alicante (Spain) in September 1999. The work presented there was really innovative as well as good for the knowledge of the situation of polymer analysis in general. The reason of the publication of papers from that Workshop is the intention of editors for getting the maximum diffusion of the lectures as well as giving the young researchers a good chance to present their work. We hope to be succeeded on our main goal.

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Chapter 1

BIODEGRADATION OF POLYMERS: KINETICAL ASPECTS

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INTRODUCTION

The problem of producing special synthetic polymers to be used in contact with biological media first arose in the 1950s. It was created by the need of biology and medicine for synthetic polymeric materials to be used in the manufacture of various endoprostheses. This led to the development of a completely new area in technology: the production of polymers for medical applications [1-3].

Both in the case of testing existing polymers for medical applications and when modified materials are used, the minimization of the undesirable interactions between the material and the tissue of the living organism in implants of short or long duration as a function of the properties of the material is the most urgent problem.

Bioengineers who produce articles from these materials and doctors who use them in practice are interested in the biocompatibility of the polymers and in the retention time of desirable properties (mechanical, diffusional, optical, etc.).

When in contact with biologically active media (tissues) most polymers undergo two mutually related processes: degradation under the influence of the biologically active substances and interaction of the degradation products with the biological media responsible for the degradation (i.e., an influence of the metabolic and of the conjugation products on the life processes of the organism). The study of the macrokinetic aspects of these processes ultimately allows the prediction of the lifetime of the polymer under stated operating conditions.

REACTION OF THE ORGANISM ON THE POLYMER

1. Initial Reaction of the Organism.

A polymer in contact with the tissues of a living organism not only undergoes changes in its physical and chemical properties but also stimulates (as a foreign body) a corresponding reaction from the organism. As a result of the damage produced in the tissues, the implantation of polymers into a living organism is accompanied by an inflammatory process [4]. This reaction consists, in the first stage, of damage to the tissues, a decrease in pO_2 and in pH, a decrease in the concentration of potassium, sodium, calcium, and magnesium ions, an accumulation of physiologically active substances resulting from the breakdown of the cell membranes and the evolution of circulating lymphocytes, and an increased accessibility of the microvessels.

In the second stage of the inflammatory process different forms of cell (neutrophils, monocytes, basophils, macrophages, etc.) are accumulated as a result of the directed motion of cells towards the inflammation region stimulated by the chemical irritants (chemotaxis). Chemical irritants include the physiologically active substances released by damaged cells and tissues (Table 1), and also substances released by the polymeric materials (degradation products, residues of catalysts, stabilizers, fillers, etc.) [5]. Thus in this stage polymers can actively influence the progress of the inflammatory process.

If the process is not complicated by the onset of infections this stage of the inflammatory process is completed (on average) in three days, and the neutrophils (which were the predominant species initially) are replaced by monocytes, plasma cells, lymphocytes, and macrophages [6].

2. Formation of Capsules Round the Polymer

After 4 to 5 days of contact the macrophages begin to predominate round the polymer, but lymphocytes, plasma cells, and fibroblasts are also present [7].

If the polymer is biocompatible the number of macrophages decreases steeply after 1 or 2 weeks, and the fibroblasts form a collagen-like capsule on the surface of the polymer (Fig. 1). If the polymer is only partially biocompatible the macrophages can survive for longer times round the implanted polymer, and under these conditions giant cells can be formed [8].

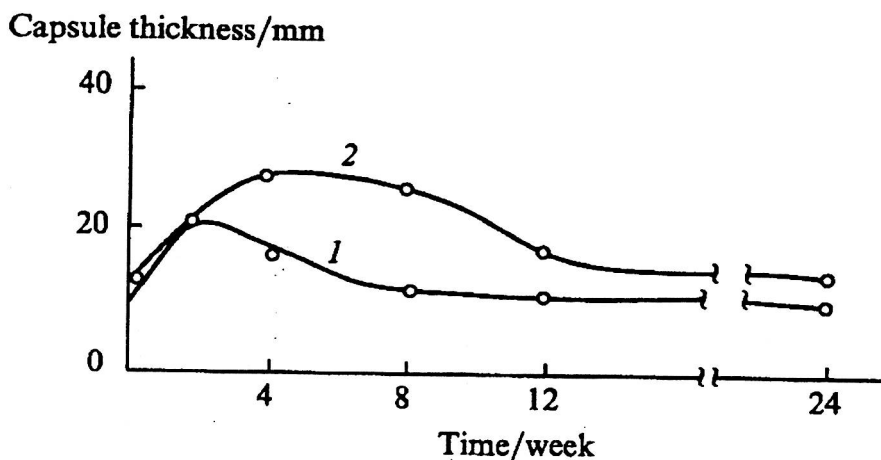


Figure 1. Plots of the capsule thickness against implantation time [14].
(1) Polydimethylsiloxane; (2) polystyrene coated with polydimethylsiloxane.

Table 1. Physiologically active substances contributing to inflammation reactions.

Substance	Chemical nature	Origin	Special features of the reaction
Histamine	β -Imidazolyl-ethylamine	Fat cells, basophils, thrombocytes	Increased penetrability of the microvessels
Serotonin	5-Oxytryptane	Fat cells, basophils, thrombocytes	Increased penetrability of the microvessels
Catecholamines	Noradrenaline	Fat cells, basophils, thrombocytes	Increased penetrability of the microvessels
Kinins	Polypeptides (metabolic products)	Plasma	Increased penetrability of the microvessels, pathogenic reactions
Hageman factor	Protease, globulin	Plasma	Increased penetrability of the microvessels, pathogenic reactions
Plasmin	Protease	Plasma	Increased penetrability of the microvessels, pathogenic reactions
Complement	Plasma proteins and metabolic products	Endothelium cells	Chemotaxis
Leukokinins	Polypeptides	Leucocytes	Increased penetrability of the microvessels
Lysosomal enzymes	Within-cell proteins	Neutrophils, macrophages, fat cells	Chemotaxis, phagocytosis
Lymphokinins	Within-cell proteins	Lymphocytes	Immune reactions in the organism
Low-molecular regulators	Prostaglandins	Precursors present in all cells except erythrocytes	Increased penetrability of the microvessels, modulation of the inflammation, chemotaxis

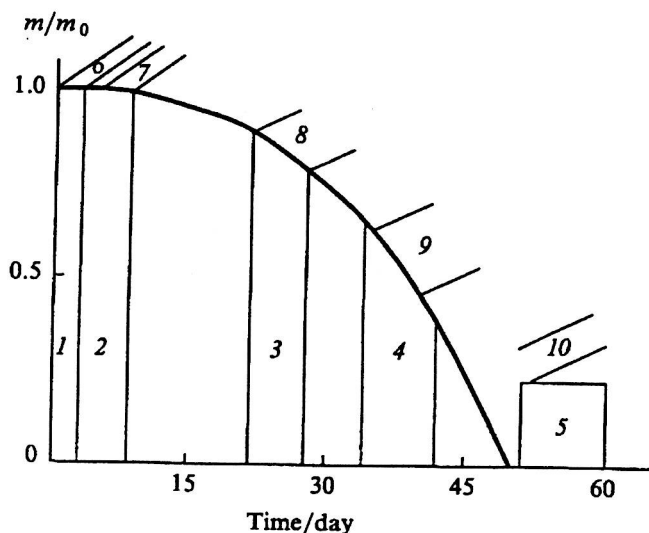


Figure 2. Dependence of the mass of the implant on time, and on its histomorphological surroundings during the degradation of a polymer with satisfactory compatibility (polyglactin).

(1) Macrophages round the implant; (2) macrophages containing phagocytes, fibroblasts; (3) giant cells and active macrophages; (4) macrophages and giant cells surrounding the polymer filaments with mitochondrial regions; (5) macrophages and giant cells surrounding the residues of the polymer fragments; (6) reaction on the polymer structure; (7) reaction to an operational trauma and healing, synthesis of collagen; (8) participation in the hydrolysis of the implant through the enzyme system; (9) participation of the cells in the metabolic process (increase in the respiratory functions); (10) complete resorption.

The thickness of the capsule depends on the biocompatibility of the polymer [9]. If the species responsible for the inflammation (for example, the degradation products) are produced at steady state we observe a chronic inflammation leading, after a long period, to the complete degradation of the polymer.

Being the main phagocyte cells of the organism, the macrophages contribute actively to the degradation of most polymers. The macrophages contain lysosomes, which act as the "alimentary canal" of the cell [10]. The lysosomal enzymes, mainly hydrolases, can destroy the polymer (phagocytosis) by two mechanisms. The first is the exocytosis, in which liposomal enzymes are produced by the macrophage and the degradation takes place on the polymer surface. The second is the endocytosis, in which the degradation process within the cell is preceded by the absorption of polymer particles by the macrophages. For example, Winter [11] detected metal particles within the macrophages by spectroscopic methods. The dynamics of the morphological changes taking place on the surface of the polymer after implantation are illustrated in Fig. 2.

BIOCOMPATIBILITY OF POLYMERS

1. General Definitions

We shall examine in general terms the response of the organism to a polymer after implantation, with special emphasis on the problems of biocompatibility.

When implanted into the organism, polymers used in medical applications can fulfill special prosthetic or other functions (diagnostic, surgical or therapeutic) without undesirable interactions with the surrounding tissues or with the organism as a whole. At present we have no clear

definition of biocompatibility, since there are no material parameters or biological tests which could be used as a quantitative measurement of this property in polymers [12]. Biocompatibility means that the polymer can exist in contact with blood and enzymes without undergoing degradation or causing thrombosis, breakdown of the tissues, or harmful immunological, toxicological, or allergenic effects.

Pure polymers with a high molecular mass usually have low toxicity [13]. The degradation of the macromolecules produces fragments of lower molecular mass, down to the final degradation products (which could be the pure monomers). We know that the toxicity of the polymers in a given homologous series is inversely proportional to their molecular mass [13].

Depending on the rate of degradation of the polymeric implant in the biological medium, its biocompatibility is found to change. In general the biocompatibility of the polymeric material is defined not only by the initial interactions but also by its chemical stability towards the surrounding biological medium.

2. Methods of Determining the Biocompatibility

A group of papers describing quantitative tests for the biocompatibility in various series of polymers has been published in the last few years [14-18].

The *in vitro* experiments were mostly done by the "tissue culture" method, which consists of growing cells on the surface of the experimental material. Two forms of the method are known:

- (1) depositing a layer of agar on the polymer, and observing the multiplication of the cells in the medium [18-21];
- (2) depositing cells (usually fibroblasts) on the polymer and immersing the polymer inoculated with cells in the feed medium [22, 23].

The effect of the polymer on three functions of the cells is usually studied: their adhesion, survival, and multiplication.

Eleven phenomena were observed in a microscopic study [15]. Table 2 lists the polymers, the phenomena that were observed, and the doubling parameter (P_D) of the resulting fibroblast cells:

$$P_D = \frac{\lg\left(\frac{N}{N_0}\right)}{0.69} \quad (1)$$

where N is the number of cells after 48-h incubation and N_0 is the initial number of cells deposited on the polymer. From the results obtained *in vitro* all the polymers were divided into three arbitrary toxicity classes.

In vivo measurements included 6 tests of the biocompatibility:

- (1) the extent of damage to the cells;
- (2) the thickness of the capsule;
- (3) the total number of cells;
- (4) the number of multinuclear leukocytes and erythrocytes;
- (5) the number of eosinophils, lymphocytes, and giant cells;
- (6) the number of fibrocytes from mononuclear phagocytes.

Table 2. Cytotoxicity of polymers in vitro

Polymer	Phenomenon*	P _D	Toxicity class
Polypropiolactone	1	2.7	I
Polyhexamethylenediamine	1	1.9	I
Teflon	1	2.1	I
Poly lactide	1	1.6	I
Poly (ethylene oxide)	1	2.1	I
Polyacrylamide	1	2.0	I
Polycaprolactone	1	2.1	I
Polyacrylonitrile	1	2.1	I
Polyglycolide	1	2.0	I
Poly (methyl methacrylate)	1, 11	—	II
Polymethyl lactyl-2-cyanoacrylate	7, 8, 10	—	II
Polyisobutyl-2-cyanoacrylate	1	2.0	II
Poly(1,2-isopropylidene)-glycerolcyanoacrylate:			
High-molecular	4	0.4	II
Low-molecular	2, 5, 6, 7	—	III
Poly (acrylic acid)	5, 8, 9	—	III
Polydimethyl-2-cyanoacrylate	5, 7, 8	—	III

* The numbers refer to the following phenomena:

- 1 - normal adhesion and multiplication of cells;
- 2 - adhesion of a small number of cells;
- 3 - normal growth of a small number of adhering cells;
- 4 - the number of cells adhering after 48 h is negligible;
- 5 - all cells in the medium have died;
- 6 - all cells adhered to the growing surface have died after 48 h;
- 7 - cells form a clump;
- 8 - cells are not attracted to the surface;
- 9 - sudden change in pH to 5.2;
- 10 - there are some changes in the cells in the metilum after 48 h;
- 11 - the polymer adheres to the cells.

The number of cells is usually determined microscopically by defining a standard area. An eight-point scale was adopted for each test [16]. The average index for each test (**M**) was found by adding the data obtained after two implantation cycles:

$$M_{STS} = (STS_{7days} + STS_{28days}) \times 10^{-1} \quad (2)$$

where STS is the average sum of the marks on the scale for the tests.

The M_{STS} values for various polymers determined after implantation in the subcutaneous cellular tissues of the rat and their arbitrary assignment to the toxicity classes are shown in Table 3.

Table 3. Toxicity of polymers, M_{STS}

<i>Polymer</i>	M_{STS}	Toxicity class
Gelatin	2.1	I
Polyacrylamide	10.3	I
Polyglycolide	19.6	I
Polyacrylonitrile	52.0	I
Poly- α -lactide	34.7	I
Poly- β -propyllactone	69.1	I
Polycaprolactone	37.3	I
Poly (ethylene oxide)	21.8	I
Polymethyl- α -lactylcyanoacrylate	56.7	II
Poly (methyl methacrylate)	49.2	III
Polydimethyl-2-cyanoacrylate	59.1	III
Polyisobutyl-2-cyanoacrylate	49.7	III

CATALYTIC ACTIVITY OF BIOLOGICAL MEDIA

In the contact between polymers and biological media the latter usually shows a catalytic activity towards the chemically unstable bonds in the polymers. Biological media contain a large number of low-molecular and high-molecular mass substances of different chemical nature, which interact with different polymers to different extents.

An analysis of published papers showed that the following substances should be considered from the standpoint of catalytic activity: water, salts, and enzymes. The choice of these substances was made partly because they are the most widespread and partly because their catalytic activity is now generally accepted.