POLYMERS AND THEIR SYNTHESIS

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Summary

Polymers are synthetic molecular materials which contributed significantly to the impressive and revolutionary development of technology during the XXth century. This

contribution is illustrated in the section on history which also describes the evolution of this young science and the first impact of the new materials on the everyday life with mainly structural applications. Also, the introductory chapter provides basic information about molecular structure of macromolecules, including the most known exemplifications. The second chapter is devoted to describe in good details the procedures used to present for preparing polymers with a broad number of macromolecules features. These procedures include the various polymerisation reactions with mechanism and kinetic equations allowing the conversion of low molecular weight bifunctional molecules into high molecular weight macromolecules. Moreover, chemical reactions are reported which allow the conversion of one macromolecule into another one. Macromolecules, because of their peculiar structure, can assume a variety of conformations resulting from rotations around single bonds; this brings to the necessity of understanding principles which control their shape as reported in chapter 3. Because of the very high molecular weight macromolecular aggregates (polymers) behave as solids or liquids with extremely high viscosity. Thus, the study of polymers in bulk concerns, as chapter 4 reports, with the understanding of molecular and supramolecular characteristic responsible for the existence of crystalline order and the amorphous state and the respective thermal transitions. The last chapter deals with polymers applications and includes relations between structure and thermo-mechanical properties, description of the main structural applications, necessary additives and procedures for shaping polymeric materials into useful forms. Finally mention is briefly made to speciality polymers for functional applications.

1. Introduction

1.1. Historical Aspects

Professor Leo Mandelkern in his "An Introduction to Macromolecules" (The English Universities Press Ltd., London, 1972) hypothesized that our age will be named as the age of materials derived from macromolecules (Polymer age after stone, copper, bronze and iron ages).

Macromolecules have existed in natural form since life began and those such as DNA, RNA, proteins and polysaccharides play crucial roles in plant and animal life. From the earliest time, man has exploited naturally-occurring polymers as materials for providing clothing, decoration, shelter, tools, weapons, writing materials and other requirements. Successively, chemical modification of naturally-occurring macromolecules allowed to produce semi-synthetic polymer based materials.

Masticated (i.e. subjected repeatedly to high shear forces), natural rubber becomes more fluid making it easier to blend with additives and to mould (1820, Thomas Hancock). The elastic properties of natural rubber could be improved, and its tackiness eliminated, by heating with sulphur (1839, Charles Goodyear). The vulcanisation of natural rubber with large amounts of sulphur produced a hard material more commonly known as hard rubber, ebonite or vulcanite (1851, Nelson Goodyear) (see *History of Chemistry*).

Successively, cellulose nitrate, also called nitrocellulose or gun cotton, was prepared by Christian Schönbein in 1846 to be used as an explosive, and then was found to be a hard

elastic material which was soluble and could be moulded into different shapes by the application of heat and pressure. A more easily processable material, named celluloid, was prepared using camphor as the plasticizer (1870, John and Isaiah Hyatt). The "viscose process"(1892, Charles Cross, Edward Bevan and Clayton Beadle), for dissolving and then regenerating cellulose, was first used to produce viscose rayon textile fibres, and subsequently for production of cellophane film.

The first synthetic polymer to be commercialised was the phenol formaldehyde resin Bakelite (L. Backlaud 1910); while the first synthetic rubber to be manufactured, known as methyl rubber, was produced from 2,3-dimethylbutadiene in Germany during World War I as a substitute, albeit a poor one, for natural rubber.

The polymer industry growth was restricted by the considerable lack of understanding of the nature of polymers, as until 1920 the common belief was that they consisted of physically-associated aggregates of small molecules. On the contrary, Hermann Staudinger believed that polymers were composed of very large molecules containing long sequences of simple chemical units linked together by covalent bonds, and introduced the word "macromolecule". His studies of the synthesis, structure and properties of polyoxymethylene and of polystyrene left little doubt as to the validity of the macromolecular point of view. This hypothesis was further substantiated by the crystallographic studies of natural polymers reported by Hermann Mark and Kurt Meyer, and by the preparation of polyamides and polyesters of Wallace Carothers. By the early 1930s most scientists were convinced of the macromolecular structure of polymers and in the following 20 years, activity on polymers increased remarkably. The theoretical and experimental work of Paul Flory was prominent in this period and allowed the establishment of most of the fundamental principles of polymer science.

In the same period, thanks to the work of Karl Ziegler and of Giulio Natta, the catalytic polymerisation of alkenes was discovered. By the use of these systems, Natta could then demonstrate the possibility of stereospecific control bearing in some cases to macromolecules with a steric regularity comparable to that observed in several natural polymers. Following Staudinger in 1953, Ziegler and Natta were jointly awarded the Nobel Prize for Chemistry in 1963. Then in 1974 also Flory was awarded the same Prize for his contribution to make the field a mature Science.

After these discoveries, the use of different combinations and permutations derived from classical organic and inorganic chemistry have allowed to design a practical infinite number of different structures some of which have assumed great importance in our every day life. Today, the large synthetic versatility and the knowledge of structure/property relations allow the preparation of materials with exceptional and predictable mechanical, thermal and functional properties for applications in all fields of human activity.

This chapter has the double objective to explain what polymers are and how they can be synthetized. Indeed, while man-made polymers have several common features with nature-made biopolymers, they differ because can be shaped by synthetic processes to many molecular structures and properties which were not considered originally important by nature. Indeed, polymers have in our days deeply and broadly penetrated

human activity and contribute to a large extent to our welfare. The term "Polymer" usually indicates only man-made polymeric materials, however, because of the above mentioned analogies, some reference will be done also to biopolymers. From the molecular point of view both polymers and biopolymers consist of very large molecules; in most cases these molecules extend over one dimension. As their length is orders of magnitude larger than their section, are then called macromolecules (long molecules). It can happen that interchain bonding occurs among different macromolecules thus originating a very large tridimensional structure or a network. For these last giant molecules the name "megamolecules" (large molecules) can be proposed. (see: *biopolymers*)

1.2. Chemical Structure

For sake of clarity and simplicity let us examine macromolecules first. A deeper insight in their structure shows that they are charaterized by backbone with many much shorter side chains. The formation of such a long backbone is possible by interconnecting, through covalent bonds, small bifunctional molecules (monomers), which like a train carriage, can form one bond with both the proceeding and the following monomer unit (or carriage). In this way the backbone consists of many identical short segments (monomeric units). Every monomeric unit is connected through a chemical bond with both the proceeding and following unit. Clearly different side chains can be attached to these segments thus originating different structures from each type of backbone.

If all side chains of a macromolecule are the same, all the monomeric units are identical and the polymer consisting of such macromolecule (MM) will be called "homopolymer". MMs with two different monomeric units form a "copolymer"; with three a "terpolymer" is formed and so on. The particular molecular structure clearly allows the MM formed by a certain unit to assume different lengths. Therefore of even during the synthesis of a homopolymer different MMs can be obtained depending on the number of monomeric units in the chain. In general, in a synthetic homopolymer, chains of different length can be present; therefore the molecular weight is no longer a distinctive property and is usually given as an average. The most used averages are the number average molecular weight ($\bar{M}_n$) [eq.1] and the weight average molecular weight ($\bar{M}_w$) [eq.2]. The corresponding average polymerisation degrees, $\overline{DP}_n$ and $\overline{DP}_w$, which indicate the number of unit per MM, are obtained by dividing $\bar{M}_n$ or $\bar{M}_w$ by the molecular weight of the monomeric unit M_o, respectively. The corresponding relations are reported below:

$$\bar{M}_n = \frac{\Sigma_i \, n_i \, M_i}{\Sigma_i \, n_i} = \frac{\Sigma_i \, w_i}{\Sigma n_i} = \frac{total \; weight}{number \; of \; MM} \tag{1}$$

$$\bar{M}_w = \frac{\Sigma_i \, w_i \, M_i}{\Sigma_i w_i} = \frac{\Sigma_i \, n_i \, M_i^{\,2}}{total \; weight} \tag{2}$$

where i is the index for species having the same molecular weight M_i, n is the number of MM and w the weight; usually in the number of MMs is reported as moles.

The above definitions of molecular weight and polymerisation degree do not consider that the macromolecule must have two terminal groups; these are indeed a negligible amount by weight if the molecular weight of the MM is large. Indeed, if M indicates a monomeric unit, the general formula of a generic MM derived from this unit can be reported as:

$$X \left(M \right)_n Y$$
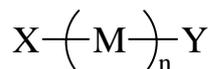
where X and Y are the end (terminal groups).

Clearly, their weight can be reasonably neglected for polymers (n > 50) and has a certain effect only in case of oligomers (low molecular weight MM with n ~ 10).
In copolymers, and clearly in more complex multipolymers, the concentration and distribution of the different units must be considered. Thus for a copolymer, whose MM consists of two different units M_1 and M_2, a practically infinite number of MMs can be designed by varying the M_1/M_2 ratio and the possible distribution along the chain. Also different copolymers can be obtained by mixing MMs with different structures. Actually, the system of possible interest are limited to few cases of MMs and then copolymers.

Thus the following MMs are named as with alternating (A), random (B), block (C) and comblike (D) distribution:

$M_1M_2M_1M_2M_1M_2M_1M_2M_1M_2$ (A)

$M_1M_2M_1M_1M_2M_1M_2M_2M_2M_1$ (B)

$M_1M_1M_1M_1M_1M_2M_2M_2M_2M_2$ (C)

$M_1M_1M_1M_1 \text{——} M_1M_1M_1 \text{——}$
$\qquad | \qquad\qquad\quad |$
$\qquad M_2 \qquad\qquad\quad M_2$
$\qquad | \qquad\qquad\quad |$
$\qquad M_2 \qquad\qquad\quad M_2$
$\qquad \vdots$

           (D)
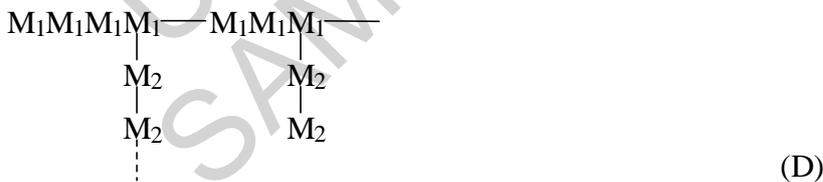
The corresponding copolymers will be called alternating, block and grafted copolymers, if consisting of MMs with structure A, C and D respectively. The random copolymer contains a mixture of MMs with all possible distributions obtainable by distributing units M_1 and M_2 without any rule along single chains and among different chains.

1.3. Stereochemical Structure

Most monomeric units contain one or more stereogenic centres. This bring to an additional differentiation of the possible macromolecules of a homopolymer. In the most simple case of a single stereogenic centre, two different monomeric units can be present in the MM of the homopolymer. As in the case of the copolymerisation a few limiting cases have practical importance as described from the different distribution in each MM of the two configurationally different units. If the stereogenic centre is an asymmetric carbon atom the following distinct situations are usually considered:

1) all units of each MM have the same configuration. The corresponding polymer is called "isotactic". In this case two situations can occur: a) all MMs are identical b) two type of MMs exist with units of one or the other configuration of the monomeric units;
2) units of different configuration alternate along the MM. The polymer is called "syndiotactic".
3) the two types of units are randomly distributed both intra- and inter MMs. The polymer is called atactic (Figure 1).

Additional situations are possible with monomers and monomeric units containing more than stereochemical center which give rise to larger number of stereochemical macromolecules.

a) isotactic polypropylene
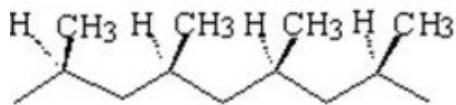
Fischer projection

b) syndiotactic polypropylene
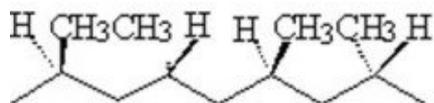
Fischer projection

c) isotactic poly-(s)-(α-alanine)
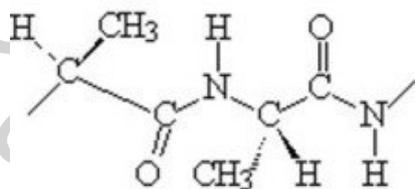
Fischer projection

Figure 1. Examples of stereoregular structures

1.4. Typical Examples

Polymerisation at the multiple carbon-carbon bond leads to macromolecules having a backbone of carbon atoms while variability may be generated by side chains. These

polymers are generally nominated by the name of the monomer with the prefix "poly". This nomenclature is not formally correct from the molecular point of view, but is used universally. A more correct nomenclature was elaborated by IUPAC but its use is very limited.

The polymerisation of ethylene to polyethylene provides a typical example (Scheme 1) of this type of MMs.


$$n\ CH_2{=}CH_2 \longrightarrow \left(CH_2{-}CH_2\right)_n$$

ethylene   polyethylene
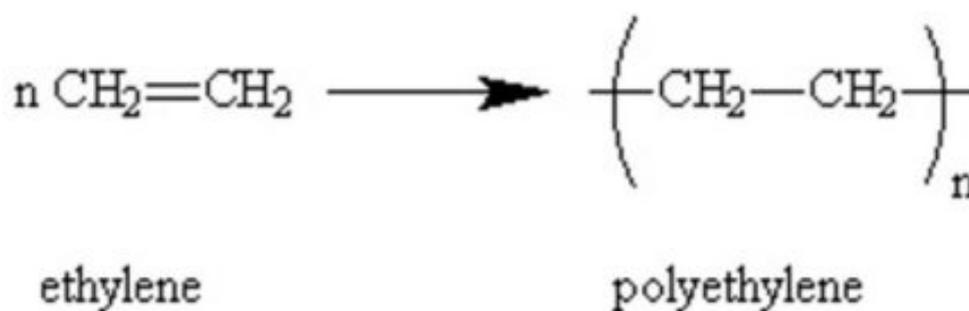
Scheme 1. Polymerisation of ethylene

The macromolecule is represented by the monomeric unit in brackets, with the suffix n indicating that the MM contains n identical units one after the other. The end groups are generally not indicated and their nature depends on the polymerisation process, being initiator (catalyst) residue and products derived from the monomer. Because n is very large some structural defects are possible. In case of polyethylene, these are due to insertion in the backbone of α–olefin formed through parallel dimerization and oligomerization reactions or to branching reaction in the free radical polymerisation at high temperature (250°C) and pressure (>1000bar).

These defects increase the number of –CH$_3$ per MM (a perfectly linear chain cannot have more then two CH$_3$, both at the chain end).

The number of these side groups can be increased by copolymerisation of ethylene with α-olefins with formation of linear and branched units along each MM (Scheme 2).

$$CH_2{=}CH_2 + CH_2{=}CH_{\underset{CH_3}{|}} \longrightarrow \left(CH_2{-}CH_2\right)\left(CH_2{-}CH_{\underset{CH_3}{|}}\right)$$

ethylene   propylene   random ethylene/propylene copolymer
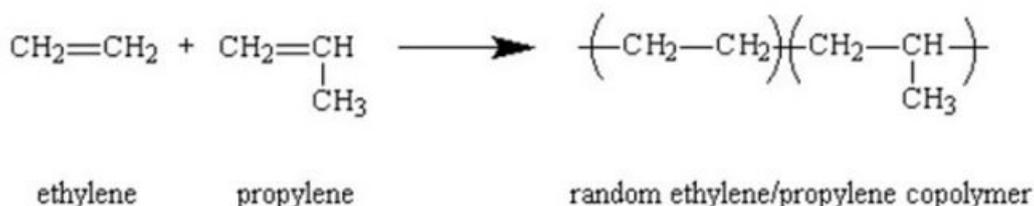
Scheme 2. Copolymerisation of ethylene with propylene

Homopolymers of α-olefins are characterized by the two different steric possibilities of enantiomeric units as the tertiary carbon atom is formally asymmetric (see Figure 1).

Examples of the most common structures of more diffuse polymers derived from monomers containing a double bond are reported in Table 1.

Monomer		Polymerisation process	Polymer	
Name	Structure		Name	Structure
Ethylene	$CH_2{=}CH_2$	Catalytic	Polyethylene	$-CH_2-CH_2-$
Propylene	$CH_2{=}CH$ $\quad$ | $\quad CH_3$	Catalytic	Polypropylene	$-CH_2-CH-$ $\qquad$ | $\qquad CH_3$
Vinyl chloride	$CH_2{=}CH$ $\quad$ | $\quad Cl$	Free radical	Polyvinyl-chloride	$-CH_2-CH-$ $\qquad$ | $\qquad Cl$
Styrene	$CH_2{=}CH$ $\quad$ | $\quad Ph$	Free radical, anionic and catalytic	Polystyrene	$-CH_2-CH-$ $\qquad$ | $\qquad Ph$
Acrylonitrile	$CH_2{=}CH$ $\quad$ | $\quad CN$	Free radical	Polyacrylonitrile	$-CH_2-CH-$ $\qquad$ | $\qquad CN$
Methyl methacrylate	$CH_2{=}CCH_3$ $\qquad COOCH$	Free radical, anionic	Polymethyl-methacrylate	$\qquad CH_3$ $\qquad$ | $-CH_2-C-$ $\qquad$ | $\qquad COOCH_3$
Vinylacetate	$CH_2{=}CH$ $\quad$ | $\quad OCOCH$	Free radical	Polyvinylacetate	$-CH_2-CH-$ $\qquad$ | $\qquad OCOCH_3$
Tetrafluoro-ethylene	$CF_2{=}CF_2$	Free radical	Teflon	$-CF_2-CF_2-$

Table 1. Examples of polymers derived from unsaturated monomers through chain polymerisation

Other addition polymers can be obtained by ring opening polymerisation of cyclic monomers which can bring to macromolecules with heteroatoms in the main chain (Table 2).

MMs containing monomeric units structurally similar to the previous one can be obtained through polycondensation or polyaddition of bifunctional monomers (Table 3).

Monomer		Polymerisation process	Polymer	
Name	Structure		Name	Structure
Ethylenoxide	CH_2—CH_2 with O bridge	Cationic, anionic	Polyethylenoxyde (polyethylenglycol)	—(CH_2—CH_2—O)—
Propylenoxide	CH_2—$CH(CH_3)$ with O bridge	Cationic, anionic	Polypropylenoxide	—(CH_2—CH—O)— with CH_3
Caprolactone	$(CH_2)_5$< CO, O	Anionic	Polycaprolactone	—[$(CH_2)_5$—C(=O)—O]—
Caprolactame	$(CH_2)_5$—NH, CO	Anionic	Polycaprolactame (NYLON 6)	—[$(CH_2)_5$—C(=O)—N—H]—

Table 2. Examples of polymers derived from acrylic monomers trough chain polymerisation

Monomers (Names + structures)	Polymer (Names + structures)
Ethylenglycol + terephthalic acid $HOCH_2$—CH_2—OH + $HOOC$—⟨benzene ring⟩—$COOH$	Polyethylenterephthalate (PET) —O—CH_2—CH_2—O—$\overset{\displaystyle O}{\underset{\displaystyle \ }{C}}$—⟨benzene ring⟩—$\overset{\displaystyle O}{C}$—
Hexamethylendiamine + adipic acid H_2N—$(CH_2)_6$—NH_2 + $HOOC$—$(CH_2)_4$—$COOH$	Nylon 6,6 —$\underset{\displaystyle H}{N}$—$(CH_2)_6$—$\underset{\displaystyle H}{N}$—$\overset{\displaystyle O}{C}$—$(CH_2)_4$—$\overset{\displaystyle O}{C}$—
p-diaminobenzene + terephthalic acid H_2N—⟨benzene ring⟩—NH_2 + $HOOC$—⟨benzene ring⟩—$COOH$	Kevlar —$\underset{\displaystyle H}{N}$—⟨benzene ring⟩—$\underset{\displaystyle H}{N}$—$\overset{\displaystyle O}{C}$—⟨benzene ring⟩—$\overset{\displaystyle O}{C}$—
Hexamethyldiisocianate + ethylenglycol OCN—$(CH_2)_6$—NCO + HO—CH_2—CH_2—OH	Polyurethane —$\overset{\displaystyle O}{C}$—$\underset{\displaystyle H}{N}$—$(CH_2)_6$—$\underset{\displaystyle H}{N}$—$\overset{\displaystyle O}{C}$—$O$—$CH_2$—$CH_2$—$O$—
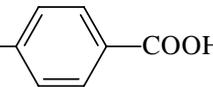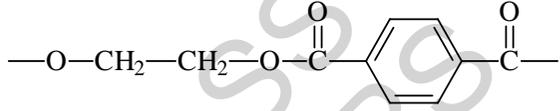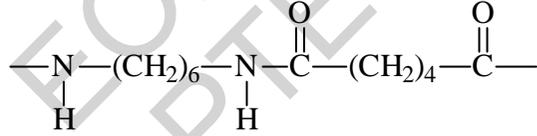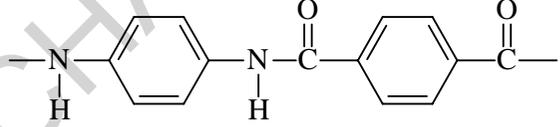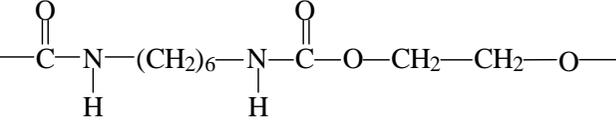
Table 3. Examples of polymer prepared by stepwise polymerisation of bifunctional monomers

The structural versatility of MMs structure can be expanded broadly by copolymerisation and varying comonomers nature and distribution (Table 4).

Name	Distrib.	Representative structure
Poly(ethylene-*co*-propylene)	random	$\left(-CH_2-CH_2-\right)\left(-CH_2-\underset{\underset{CH_3}{\mid}}{CH}-\right)$
Poly(butadiene-*co*-styrene)	random	$\left(-CH_2-\underset{\underset{CH_2}{\overset{CH}{\overset{\|}{\mid}}}}{CH}-\right)\left(-CH_2-CH-\right)\left(-CH_2-CH=CH-CH_2-\right)$
Poly(butadiene-*b*-styrene)	block	$\left[\left(-CH_2-\underset{\underset{CH_2}{\overset{CH}{\overset{\|}{\mid}}}}{CH}-\right)\left(-CH_2-CH=CH-CH_2-\right)\right]_n\left(-CH_2-\underset{Ph}{\overset{\mid}{CH}}-\right)_m$
Poly(maleic anhydride-*al*-ethylene)	alternating	$\left(-\underset{O=C}{CH}-\underset{C=O}{CH}-CH_2-CH_2-\right)$ with O bridging the two C

Table 4. Typical copolymers structure

2. The Synthesis of Macromolecules

New macromolecules and then polymers can be synthetized either by a): polymerisation of monomers and b): chemical modification of monomeric unit side chain of preformed macromolecules. The former process has the main effect to increase molecular weight whereas the latter produces chemical changes of side chain structure without molecular weight variation.

2.1. From Monomers to Macromolecules

2.1.1. Features of the Polymerisation Reactions

The chemical process which converts molecules into polymer macromolecules is known as polymerisation. This process actually is the combination of different reactions which determine the feature of the product obtained depending also on the starting chemical

composition. These reactions are in general: initiation, propagation, termination and transfer reaction. Propagation is often identified as polymerisation reaction as it is responsible for the macromolecule formation. The polymerisation is an "one pot" process and therefore the synthesis of macromolecules by successive addition of monomer units in well distinct steps cannot be considered a polymerisation. For example, proteins and nucleic acids are synthetized in this way. For detailed information, the reader is referred to the corresponding chapters. Synthetic polymers are normally obtained by submitting a monomer to conditions under which the separated monomer molecules reacts to bind each other to form a MM. However, as explained in the previous section, from the same monomer we can obtain homopolymer, MMs differing in molecular weight. Therefore the product of the polymerisation may not be formed by a simple MM, but is in general a mixture of MMs having different length. The MW of the obtained MM, or better its average, depends for a given monomer on reaction conditions and reaction time as well as conversion of monomer into MMs. Therefore all polymerisation reactions are characterized by the evolution with time of both conversion and MW. These last depend in a different way on polymerisation mechanism as observed for the two typical polymerisations based respectively on a "chain" or "stepwise" mechanism. (see*: Proteins, Nucleic Acids*)

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Biographical Sketches

Francesco Ciardelli born November 5, 1937 in Pisa, Italy married with one son.

Professional carrie:r
Research Associate, University of Pisa 1960-1965, University College, London 1965;
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Professional activity
Member of the Scientific Board of three CNR Centers and one CNRS Lab.Lyon
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J. Polymer Science, 1983-1989
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New Polymeric Materials, since 1989
Polymers, since 1989
Polymers for Advanced Technologies, since 1990
J. Molecular Catalysis, since 1995.
Macromolecular Chemistry, since 1996.
Research interests:
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b. Stereospecific Polymerization and Chiral Polymers
c. Clean Industrial Processes
d. Polymer Functionalization and Blends
e. Photoresponsive and Electroconducting Polymeric Materials
f. Polymers for Coatings and Preservation of the Cultural Heritage

Publications:
260 papers on international Journals, 6 patents, 6 books

Elisa Passaglia, born January 26, 1966, in Pisa, Italy, married with one child. Elisa is a Contract researcher at Department of Chemistry and Industrial Chemistry of University of Pisa. She obtained PhD in Chemistry from University of Pisa 1993-1996

Professional Experience:
Contract with the University of Pisa
The activities were addressed on research projects for the synthesis and characterisation of fluorinated acrylic polymers as stone protectives.
1993 – 1996
PhD Activities: the research was based onto functionalization of polyolefins and reactive blending of functionalized polyolefins with nylon.
1996-2001
Contracts with the University of Pisa
The activities were addressed on research projects for the functionalizations of saturated and unsaturated polyolefins, and the compatibilization of polyolefins with polyesters, polyamides and inorganic composites like as silica layered silicate carbon black. The studies of the functionalization and composite preparation were performed by using the mixer and the extruder and the characterizations were carried out with FT-IR, NMR, DSC, DMA, TGA analysers.
Publications:
Dr. Elisa Passaglia is co-author in 60 pubblications and about 90 presentations and 6 patents.

Simona Bronco, born March 22, 1968 in Città della Pieve (Perugia) , Italy married with two children. Simona got PhD in Natural Science from Swiss Federal Institute of Technology; 1994-1996 and Doctoral Degree in Chemistry (Summa cum Laude) from University of Pisa ; November *1987 – July 1994*. Present position is a Contract researcher at National Institute for Physical Chemistry, Research Unit of Pisa. The main research interests are the development and processing of polymeric materials in the field of the Material Science.

Professional Experience:
1992 – 1995
Scientific Co-worker at the Swiss Federal Institute of Technology Zurich (CH) - I
Activities on research projects for the synthesis and characterisation of diphosphine palladium catalysts for the copolymerization of CO and propylene
1994 – 1996
PhD Activities: the research was based onto the regio- and steric control in the copolymerization of propylene and carbon monoxide in the presence of palladium catalysts
1997-1999
Contracts with the Consorzio Pisa Ricerche
The activities were addressed on research projects for the development of polymeric material for structural and functional application in tanning and finishing process. The characterizations were carried out with FT-IR, NMR, DSC, DMA, TGA analysers. "Actual position Researcher at CNR-INFM PloyLab Pisa. The activities ware dressed on the preparation and characterization of materials from natural and renewable resources for industrial applications."
Publications:
Dr. Simona Bronco is co-author in 33 publications and about 15 presentations and 2 patents.