

Hybrid particles of graphene oxide and poly(glycidyl methacrylate) brushes, optimization of reaction conditions leading to preparation of materials with tunable electrical conductivity

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Zásady pro vypracování

Řada tkání, jako např. srdeční či nervové, využívají pro komunikace mezi buňkami elektrický signál. Při navrhování náhrad je proto nezbytné tento fakt zohlednit při přípravě vhodného materiálu. Částice na bázi alotropů grafenu nachází využití v řadě kompozitních systémů z důvodu jejich výjimečných elektrických a mechanických vlastností. V současné době probíhá intenzivní výzkum těchto částic s ohledem na uplatnění v tkáňovém inženýrství. Z důvodu zlepšení dispergace a distribuce částic v matricích je vhodné povrch částic modifikovat.

Cílem práce bude syntéza a charakterizace hybridních částic oxidu grafenu (GO) pomocí kartáčů poly(glycidyl methakrylátu, PGMA) reversibilně-deaktivační radikálovou polymerací (RDRP).

Student se bude v řešební části zabývat PGMA a GO obecně. Podrobně se bude věnovat možnostem polymerací monomeru samotného, dále technikami polymerací z povrchu oxidu grafenu. Nakonec nastíní možné použití těchto hybridních materiálů. Experimentální část bude zaměřena na optimalizaci podmínek polymerace z povrchu oxidu grafenu v organickém prostředí pomocí RDRP technik s cílem zvýšení elektrické vodivosti hybridních částic. Polymerace budou sledovány pomocí nukleární magnetické rezonance a gelové permeační chromatografie. Elektrická vodivost částic bude měřena dvou nebo čtyřbodovou metodou.

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ABSTRAKT

Tato bakalářská práce se zaměřuje na přípravu a charakterizaci hybridních částic tvořených grafenovým oxidem (GO) s navázanými polymerními kartáči poly(glycidyl methakrylátu) (PGMA) metodou povrchově iniciované radikálové polymerace s přenosem atomu (SI-ATRP). Před samotným graftováním byly optimalizovány podmínky pro homopolymeraci GMA za účelem dosažení kontrolovaného růstu polymerních řetězců a minimalizace nežádoucího zesíťování epoxidových postranních skupin. Kinetika polymerace byla vyhodnocena pomocí nukleární magnetické rezonance (NMR) a gelové permeační chromatografie (GPC). Výsledné hybridní částice byly dále analyzovány termogravimetrickou analýzou (TGA), infračervenou spektroskopií (FTIR) a měřením elektrické vodivosti. Elektrická vodivost GO byla po navázání polymeru výrazně zvýšena, což je přisuzováno částečné redukci GO během polymerace. Připravené materiály vykazují potenciál pro využití v pokročilých funkčních aplikacích, kde je žádoucí řízená elektrická vodivost.

Klíčová slova: grafenový oxid, poly(glycidyl methakrylát), SI-ATRP, hybridní částice, elektrická vodivost

ABSTRACT

This bachelor thesis focuses on the preparation and characterization of hybrid particles consisting of graphene oxide (GO) grafted with poly(glycidyl methacrylate) (PGMA) brushes via surface-initiated atom transfer radical polymerization (SI-ATRP). Prior to grafting, the reaction conditions for GMA homopolymerization were optimized to achieve controlled polymer growth and to minimize undesired crosslinking of oxirane side groups. The polymerization kinetics were evaluated using nuclear magnetic resonance (NMR) and gel permeation chromatography (GPC). The resulting hybrid particles were analyzed by thermogravimetric analysis (TGA), Fourier-transform infrared spectroscopy (FTIR), and electrical conductivity measurements. The electrical conductivity of GO was significantly enhanced after polymer grafting, which is attributed to partial reduction of GO during polymerization. The prepared materials offer potential for use in advanced functional applications where tunable conductivity is desired.

Keywords: graphene oxide, poly(glycidyl methacrylate), SI-ATRP, hybrid particles, electrical conductivity

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INTRODUCTION

In recent years, the development of functional nanomaterials has gained significant attention due to their potential applications in biomedicine, energy, and environmental technologies. Among these materials, graphene oxide (GO) stands out thanks to its high surface area, chemical reactivity, tunable conductivity, and excellent mechanical properties. Its ability to interact with various organic or inorganic species makes it a promising platform for designing advanced hybrid structures.

Another key aspect in material science is the ability to precisely control the architecture and properties of polymers. Atom Transfer Radical Polymerization (ATRP) represents a powerful tool for the synthesis of polymers with narrow molecular weight distributions and defined structures. In particular, the surface-initiated ATRP (SI-ATRP) allows the growth of polymer brushes directly from solid surfaces, such as GO sheets, thus enabling the preparation of hybrid particles with tailored properties.

This work focuses on the synthesis of GO/PGMA hybrid particles, where poly(glycidyl methacrylate) (PGMA) is grafted from the surface of GO via SI-ATRP. These materials combine the chemical versatility of GO with the functional potential of PGMA, especially in areas requiring controlled surface properties or tunable electrical conductivity. The epoxide groups present in PGMA chains can react with nucleophilic biomolecules such as amino groups in proteins, enabling potential applications in bioconjugation, surface modification, or immobilization of enzymes for biomedical purposes.

During polymerization, the oxirane rings of GMA side chains can undergo ring-opening reactions, especially under elevated temperature or in the presence of nucleophiles. This can lead to undesired crosslinking and loss of control over the polymer structure, which makes reaction condition optimization crucial for successful synthesis.

Interestingly, some ATRP ligands such as tertiary amines (e.g., PMDETA) have been shown to partially reduce GO during polymerization, leading to changes in its electronic properties, including enhanced electrical conductivity. This unintended but beneficial side effect can contribute to the functionality of the final hybrid material.

Special emphasis is placed on the optimization of polymerization conditions, with the aim of achieving good control over the process and minimizing side reactions such as oxirane crosslinking. In addition, the study explores the effect of polymer structure on the

resulting electrical conductivity of the materials, which may contribute to future applications in sensing, electronics, or biomedical engineering.

I THEORETICAL PART

1 POLY(GLYCIDYL METHACRYLATE)

Poly(glycidyl methacrylate) (PGMA) is a polymer synthesized from main monomer glycidyl methacrylate (GMA). The polymer is obtained using various polymerization methods, such as free radical polymerization, controlled radical polymerization and enzymatic polymerization. PGMA offers us remarkable functionalization potential due to the exceptional versatility of the chemistry of the epoxy groups [1]. It finds use in industrial and biological applications [2].

1.1 PGMA properties and application

PGMA exhibits relatively high thermal stability, allowing processing at elevated temperatures without significant degradation of the final polymer. It is also a polymer with good mechanical strength and stiffness and has good acid and base resistance. Therefore, it can be used for applications requiring dimensional stability and resistance to deformation [3].

Thanks to its biocompatibility and the reactivity of epoxy groups, this polymer finds applications in the biomedical and pharmaceutical fields. It is used for drug or genetic material delivery with controlled release or in tissue engineering, where it provides a suitable environment for cell growth and tissue regeneration. It is also used in nanocomposites, functional materials, surface modifications, and adhesive applications [4].

1.2 Monomer GMA

Glycidyl Methacrylate (GMA) is an ester of Methacrylic Acid and Glycidol. GMA undergoes a wide range of chemical reactions through the opening of its oxirane ring, allowing for extensive chemical modifications in the resulting polymers. Depending on the intended application, the epoxy group in GMA has been commonly used as a grafting monomer [2]. GMA reacts with various substances, such as amines, phenols, ketones, carboxylic acids, and others, enabling the formation of new chemical bonds. Even small additions of this monomer to polymer materials can ultimately enhance their mechanical, optical, and adhesive properties, as well as their chemical resistance [3].

1.3 GMA structure

GMA is characterized by a unique structure – Figure 1, containing both vinyl and epoxy groups. The vinyl group enables polymerization through radical mechanisms, while the epoxy group allows for subsequent post-modification or cross-linking [2].

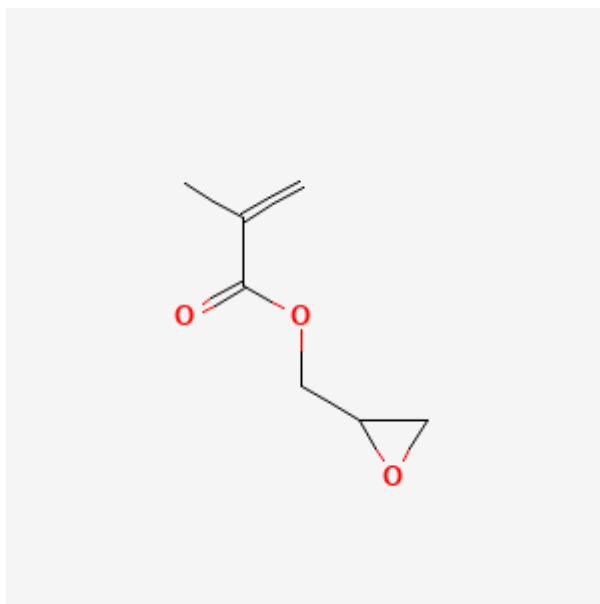


Figure 1: GMA structure [5]

Glycidyl methacrylate was selected as the monomer for grafting due to its ability to form poly(glycidyl methacrylate) brushes with reactive epoxide groups. These functionalities allow further chemical modification or bioconjugation, making PGMA a versatile component in the design of functional hybrid particles.

2 GRAPHENE OXIDE

Graphene oxide (GO) is one of the most important allotropes of graphene. Other allotropes include for example bare graphene, graphite or reduced graphene oxide (rGO) [6; 7].

The basic structure of GO consists of sp^2 hybridized carbon atoms arranged in a hexagonal lattice structure. Additionally it contains oxygen-containing functional groups attached to the carbon hexagonal lattice structure. Epoxy groups (-O-) are bonded to carbon atoms within the layer, while hydroxyl groups (-OH) are also attached to carbons on the surface of the graphene sheet. In contrast, carbonyl groups (-C=O) are located at the edges of the layers, and carboxyl groups (-COOH) are primarily attached to the layer edges [8] – Figure 2.

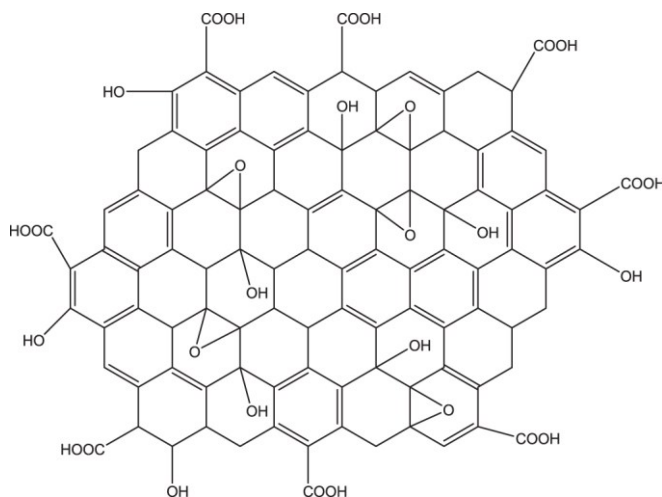


Figure 2: GO structure [9]

Due to the presence of oxygen-containing functional groups, GO exhibits different properties compared to pure graphene. It does not absorb visible light, has much lower electrical conductivity, it's hydrophilic and exhibits significantly greater chemical reactivity [10]. GO enables diverse chemical modifications by attaching various functional groups to its surface, allowing for the adjustment of optical transparency, electrical conductivity and thermal properties [10].

The starting material for graphene oxide preparation is graphite. Through oxidation with strong oxidizing agents, such as strong acids and oxidizers, oxygen-containing functional groups are introduced into the carbon structure, forming graphite oxide. This graphite oxide is then exfoliated, resulting in a suspension of graphene oxide particles, which exhibits a yellow coloration [10; 11] – Figure 3.

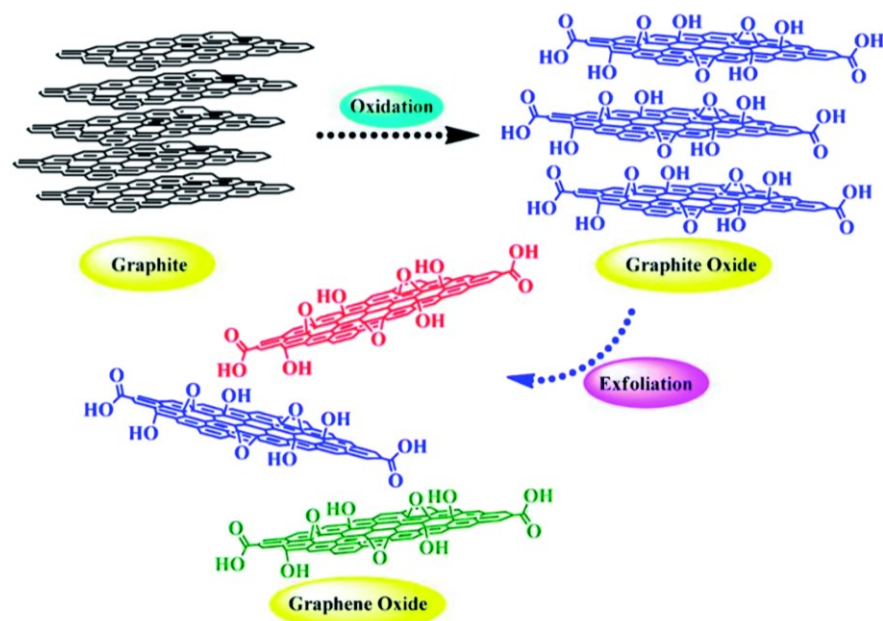


Figure 3: Preparation of graphene oxide [11]

For the first time in history the graphene oxide was identified in 1859 by professor Brodie from the University of Oxford. Over the course of the following years, Brodie's method was modified by various researchers leading to the development of several well-known methods for graphene oxide preparation such as Staudenmaier, Hofmann, Hummers, and Tour method [11; 8]. These well-known methods have been frequently adapted to meet the specific requirements of individual researchers. Numerous studies have reported various approaches to the synthesis and preparation of graphene oxide with differences in the resulting structure depending on the chosen method [12].

2.1 Structure of graphene oxide

In addition to the fundamental structure of GO, other structural variations have also been identified – Figure 4.

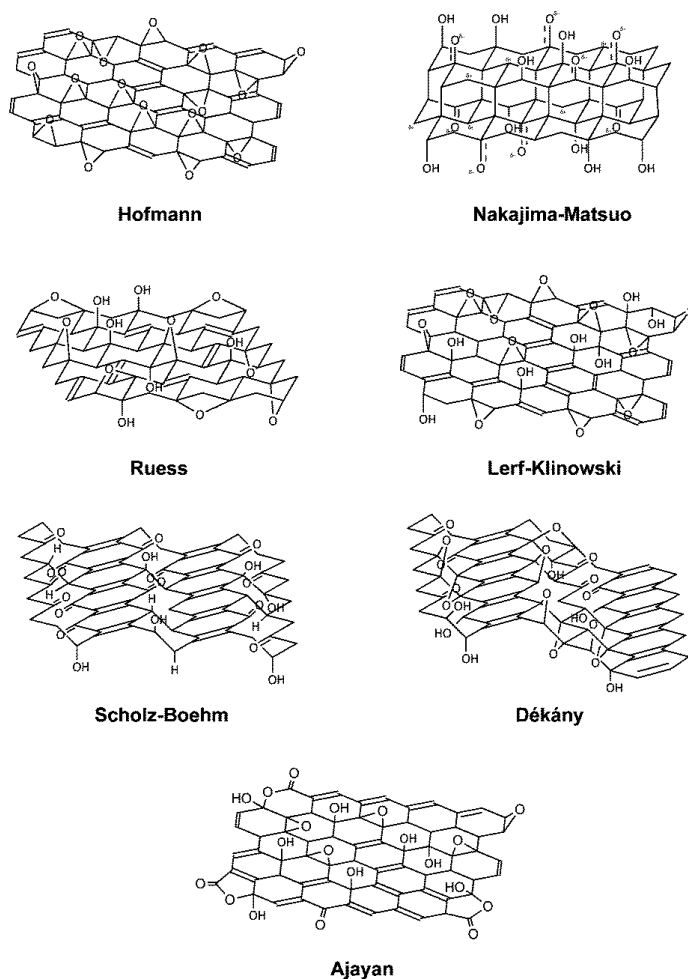


Figure 4: Different models of GO structure [8]

The first proposed model of graphite oxide by Hofmann and Holst suggested the presence of repeating 1,2-epoxide units across the entire basal plane of graphene. Compared to the first model, Ruess described the structure in which the basal planes were sp^3 hybridized. This model consisted of 1,3-epoxide and hydroxyl groups. Scholz and Boehm modified previous approaches by proposing a model consisting exclusively of hydroxyl and ketone groups. Described structure by Nakajima and Matsuo suggests that GO is similar to graphite intercalation compounds. Lorf-Klinowski model proposed a model consisting of two distinct regions – aromatic and aliphatic. This model assumes that epoxide and hydroxyl groups populate the basal plane, while carboxyl and hydroxyl groups are located at the layer edges.

Dékány proposed a structure based on multiple analytical techniques and his model, derived from the Ruess and Scholz-Boehm models, divides GO into two primary domains – cyclohexane chairs and corrugated hexagonal ribbons.

Ajayan and his team identified signals corresponding to lactols, specifically 2-hydroxynaphthoic acid anhydrides and 1,3-dihydroxyxanthenes. Their proposed functional group composition included hydroxyl, epoxide, lactol, ester, and ketone carbonyl groups [8].

2.2 Graphene, graphite

Graphene is a two-dimensional material consisting of a single layer of carbon atoms arranged in a hexagonal honeycomb lattice. It serves as the fundamental building block of various carbon allotropes, including graphite, carbon nanotubes, and fullerenes. Graphene holds the distinction of being the most strongest material ever measured [13].

Graphene exhibits a range of unique physical and chemical properties, making it one of the most significant materials in modern science and technology. This carbon allotrope possesses an exceptionally high specific surface area of 2630 m²/g. Graphene exhibits remarkable thermal conductivity exceeding 5000 W/m·K, along with outstanding electrical conductivity due to the nearly resistance-free movement of electrons. Another outstanding property is its significant mechanical strength, with a tensile strength of approximately 130 GPa, while maintaining significant flexibility. Additionally, graphene demonstrates notable chemical reactivity and can undergo surface modifications for various applications [14; 15].

There are various methods for synthesizing graphene or extracting graphene layers, depending on the desired final properties such as the number of layers, size, or purity. These methods can generally be divided into two main categories [13; 14]

The first is top-down processes, which are based on the exfoliation of starting materials – most commonly graphite or its derivative graphene oxide [13]. The advantage of these methods lies in their simplicity and ability to produce large quantities of graphene. However the resulting material may exhibit lower quality. These methods include mechanical, chemical, and electrochemical exfoliation, as well as the unzipping of carbon nanotubes [14].

The second category includes bottom-up processes, in which graphene is synthesized by assembling carbon atoms into a regular hexagonal lattice. Compared to the first category,

these methods offer a higher degree of control over the quality of the resulting material. Bottom-up approaches include chemical synthesis, epitaxial growth, microwave synthesis and chemical vapor deposition (CVD) [14; 16].

Its exceptional properties make it suitable for a wide range of advanced applications in various fields such as electronics and nanoelectronics, energy storage and conversion, thermal management, sensors, composites and coatings, transparent conductors, biomedical applications [15].

Graphite is one of the natural crystalline allotropes of carbon which consists of layers of carbon atoms arranged in a hexagonal lattice. Graphite is generally regarded as being composed of a large number of stacked graphene layers. Two types of bonds are present between these layers. The graphene layers are bound by weak van der Waals forces, with an interlayer spacing of approximately 0.341 nm. This weak interaction facilitates the exfoliation of graphite into individual graphene sheets. In contrast, within each layer, the carbon atoms are connected by strong covalent bonds with a bond length of 0.142 nm – Figure 5.

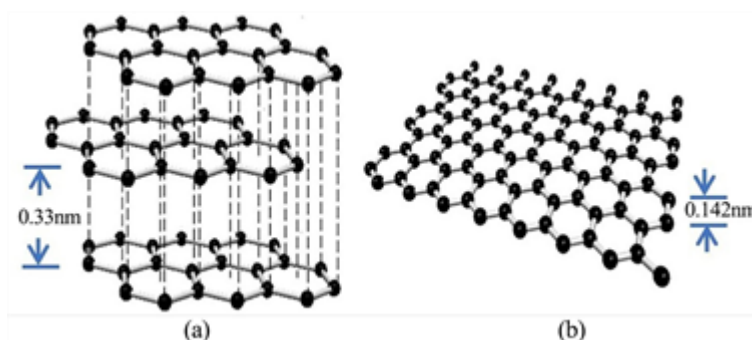


Figure 5: Structure of (a) graphite (b) single layer of graphene [13]

This combination of strong intralayer and weak interlayer interactions makes graphite an anisotropic material – it exhibits different properties in different directions. Graphite exhibits a soft texture due to the weak bonding between its carbon atom layers, in contrast to the strong covalent bonds holding the atoms together within each layer [14].

Graphite is found in metamorphic rocks, often formed from the metamorphism of organic material like coal under high pressure and temperature. Graphite comes in several forms with different characteristics and the main classification is into natural and synthetic types [17].

Natural graphite is mined from geological deposits and is categorized into three subtypes based on its crystal morphology. First of these subtypes is flake graphite, which is found in metamorphic rocks and it appears as flat, plate-like particles. It is characterized by high purity after processing, good conductivity and lubricity. It is used in batteries, lubricants and refractories. The second subtype is amorphous graphite which is finer, less crystalline than flake graphite and found in coal seams. It has lower purity and conductivity than the first subtype and is used in the field of brake linings and also lubricants. The last subtype of natural graphite is vein graphite, characterized by its rarity, high-purity, excellent electrical and thermal conductivity. It is used in high-performance applications [18].

Synthetic graphite is artificially created by high-temperature processing of materials that are very rich in carbon. These materials include, for example, petroleum coke or coal tar pitch. The production process is called graphitization. Graphite can be produced in a very pure form by this process and has excellent thermal and electrical conductivity. It is used in the manufacture of electrodes, aerospace components, nuclear reactors, and high-performance batteries [14; 18].

2.3 Application of graphene oxide

Graphene oxide (GO) is used in many industries thanks to its properties. These include its large surface area and extensive π -conjugated structure, which allows it to bind different types of proteins depending on the desired application. Furthermore, its structure also contains a large number of oxygen atoms, which makes it a hydrophilic material. Last but not least, it also has high mechanical resistance and adjustable electrical conductivity.

2.3.1 Power and energy storage

It is the above properties of GO that enable its effective use in the energy sector, specifically in the production of supercapacitors. The combination of GO with conductive materials such as carbon nanotubes, conductive polymers or transition metal oxides leads to an increase in electrochemical capacity and electrode stability. These composites contribute to cycling stability and maintain the increased capacitance for several thousand cycles. GO also enhances the wettability of the electrodes, thereby accelerating the ion transfer in the electrochemical system [19]. GO is also applied in this sector in battery production, particularly in lithium-ion, lithium-sulphur batteries and others. In Li-ion batteries, GO serves as a cathode coating and this results in better conductivity, higher cathode

performance and also suppresses aggregation of the active material during use. It can also be used as an anode material, especially together with Si, or as corrosion protection, thereby increasing the lifetime of the battery [19].

Furthermore, this promising material finds applications in energy conversion devices such as fuel cells and water electrolyzers. These systems use ion-conducting membranes and electrodes and this is where graphene oxide can be used. In fuel cells, a proton-exchange membrane (PEM) is used, which only transmits protons and not electrons. Due to its layered structure and functional oxygen groups, GO allows the movement of protons in its individual layers. This ability, together with its hydrophilic nature, makes the graphene oxide-based membrane an effective alternative to the aforementioned PEM membranes [20].

Other applications of graphene oxide in this area include applications in sodium batteries, flow batteries, methanol fuel cells, dielectric materials and capacitors, electrochemical sensors, and more [20].

2.3.2 Environment protection and water filtration

In the environmental and water filtration industry, graphene oxide is mainly characterized by its large surface area, which provides a strong adsorption capacity. GO therefore serves as an excellent sorbent, especially for emerging contaminants such as pharmaceuticals, pesticides, dyes and cosmetic impurities. Another advantage for the industry is the possibility of GO functionalization, where the surface is modified to improve adsorption, selectivity or to interact specifically with certain contaminants. GO-based adsorbents can also be reutilized, reducing waste and cost [21].

According to research, GO can also be classified in membrane systems where its structure gives the membrane unique properties such as high permeability to water, excellent selectivity for ions and organic molecules, ultra-thin membrane layer or the possibility of adjusting the interlayer distance. With these properties, these graphene oxide membranes are included in modern water treatment applications and also have great prospects for use in seawater desalination [22]. This type of use of graphene oxide is still in the research part, but is already proving to be very effective.

2.3.3 Catalysis and photocatalysis

Again due to its already mentioned properties, graphene oxide is suitable for use in heterogeneous catalysis and photocatalysis. In the process of photocatalysis, chemical decomposition of substances occurs in the presence of a photocatalyst and light radiation. GO actively participates in this process by capturing and transferring photogenerated electrons, thereby enhancing the process. Graphene oxide also helps to narrow the band gap in other semiconductors, resulting in a narrowing of the energy gap between the valence and conduction bands, and electrons can more easily pass into the conduction band, making it easier for the material to conduct an electric current [23].

GO is used either as a photocatalyst by itself or more commonly used as a co-catalyst in combination with metal oxides such as TiO_2 or ZnO . In these hybrid systems, Ti-O-C and Zn-O-C bonds are formed and it is due to these interactions that the aforementioned band gap narrowing occurs. Due to the presence of reactive functional groups and in particular the π - π interactions, GO can also be used as a stand-alone catalyst. When irradiated with UV or visible light, it can generate radicals and decompose organic substances without combining with metal oxides [23].

Another important application of GO in photocatalytic systems is as a template for the construction of two-dimensional growth nanostructures. In the synthesis of photocatalytic composites, graphene oxide can serve either as a support layer for heterogeneous nucleation, where the crystallization of these composites occurs directly on its layer. Or as a routing template, which helps to form the regular morphology of the nanostructures in question and, last but not least, as a dispersion stabilizer that promotes uniform surface coverage. Such controlled growth has significant advantages, with the resulting photocatalytic structures having a larger specific surface area where more reactions or adsorption can occur, as well as enhanced structural and mechanical stability. This type of use is applied for use in multilayer composites, hydrogels or 3D aerogels [24].

2.3.4 Construction and composite materials

The use of graphene oxide has also proven itself in the construction industry. Here GO acts as a nano-reinforcement in the cement matrix. This combination has been found to have a great effect on the desired properties. Compressive, flexural and tensile strengths are significantly improved already at low concentrations. GO also accelerates the hydration of

the cement and refines the pore structure, thus permeability is reduced and resistance to chemicals is increased [25; 26].

Due to its electrically conductive properties, graphene oxide can act as a sensor interface with which we are able to monitor stress, vibration and micro-crack formation. By combining with materials such as metal fibres, microencapsulants etc., self-repairing composites are formed. These composites work on the principle of secondary hydration or release of the repair agent. GO plays a role in water distribution, controlling the growth of hydration products or in strengthening the bonds around the resulting crack. This can significantly advance durability even under severe application conditions [26].

2.3.5 Tissue engineering

Graphene oxide (GO) has emerged as a promising nanomaterial for tissue engineering due to its unique combination of mechanical strength, surface functionality, and biological responsiveness. It enhances the performance of scaffolds by contributing to their structural stability and promoting beneficial cell–material interactions. Thanks to its antibacterial activity and ability to bind and gradually release bioactive molecules, GO offers a multifunctional platform suitable for various types of tissues, including neural, muscular, cartilage, bone and cardiac [27].

As a functional component in scaffold systems, graphene oxide provides structural support and creates a favorable microenvironment for cell adhesion, proliferation, and differentiation. Thanks to its high specific surface area, functional oxygen-containing groups, and excellent processability in composites, GO enhances the bioactivity of scaffolds by improving surface functionality and promotes stem cell differentiation [28]. A particularly valuable property of both GO and its reduced form (rGO) is their electrical conductivity, which enables active stimulation of electrically responsive tissues such as cardiac muscle, nerves, and skeletal muscle. Consequently, GO and rGO are widely incorporated into conductive scaffolds that not only support cell viability but also enhance tissue regeneration via bioelectrical signaling [29].

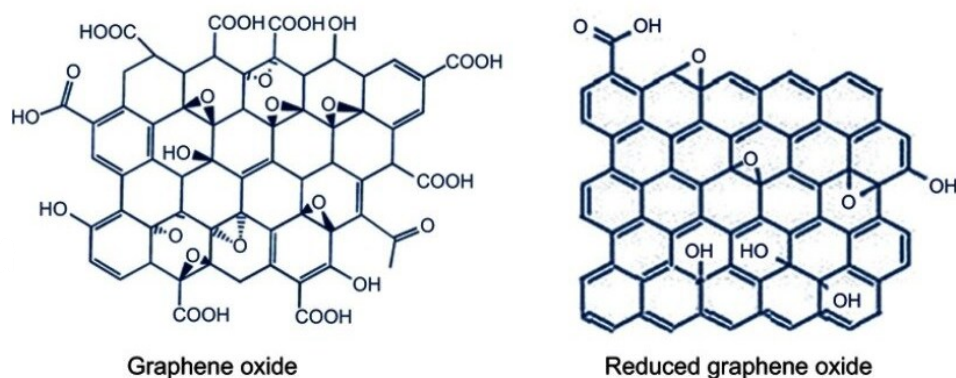


Figure 6: Structure of a) GO; b) rGO [29]

GO and rGO were also tested in the regeneration of central (CNS) and peripheral (PNS) nervous tissue. In this application, GO scaffolds form an electrically conductive interface, thereby supporting the electrophysiological environment of neurons, serving as a platform for growth and orientation of neural processes, and most importantly, as a carrier of neurotrophic factors that accelerate regeneration [27].

GO has also been used in the construction of scaffolds for bone and cartilage tissue. In bone engineering, GO is used as a reinforcing component of scaffolds, supports osteoblastic activity due to its bioactive surface and acts as a carrier for osteogenic factors [29]. The ability of graphene oxide to bind calcium and form nucleation sites for hydroxyapatite promotes mineralization. The GO surface promotes adhesion and growth of osteoblasts and stem cells. Specific results indicate that GO at concentrations of 0.1-1.0 % in composites exhibits an ideal ratio between bioactivity and cytocompatibility. In vivo tests on bone defect models show accelerated healing and increased volume of newly formed bone [28]. Like bone, cartilage tissue is a poorly vascularized tissue, which complicates its spontaneous regeneration.

In cartilage regeneration it was revealed that, GO plays a role as a carrier of growth factors, a platform for chondrogenic differentiation and a reinforcing phase of the scaffold. GO stabilizes growth factors, protects them from degradation and ensures controlled release. Graphene oxide also contributes to the protection of chondrocytes from oxidative stress, thereby improving survival in hypoxic conditions of articular cartilage [28]. As with bone and cartilage tissue, it is known from the literature that scaffolds enriched with GO show higher production of newly formed tissue, which was demonstrated in both in vitro and in vivo tests [28].

Due to its layered structure and large specific surface area, GO is applied as a platform for controlled delivery of drugs, peptides, proteins and growth factors. The oxygen functional groups localized on its structure allow both covalent and non-covalent binding of bioactive molecules, and the layered structure ensures adsorption of substances between the layers. This controlled release application is used in the local administration of growth factors, controlled release of anti-inflammatory drugs or can also be used for targeted therapy in the area of wound healing, treatment of inflammation [27]. It can also be used for the application of a so-called intelligent scaffold, which applies the drug as needed and can respond to changes in the environment [27; 28].

Graphene oxide has also shown promise in recent years as a material for skeletal muscle regeneration. Primarily due to its ability to promote myogenic differentiation and angiogenesis, a term for the re-formation of blood capillaries in a damaged tissue bed. These abilities are due to the exocrine secretion of vascular endothelial growth factor (VEGF), which contributes to the formation of new muscle tissue and its adequate vascular supply [30]. Increasing myogenesis is also aided by the ability to conduct electricity and use new muscle tissue for electrical stimulation [31]. This application can be very effective in cases of extensive muscle loss where natural regeneration is limited.

In this work, graphene oxide is used as a solid support for polymer grafting due to its large specific surface area, functional groups, and tunable properties. Its chemical reactivity enables surface functionalization, while its conductivity can be enhanced during polymerization, contributing to the functionality of the resulting hybrid material.

3 GRAPHENE OXIDE HYBRID PARTICLES

Although GO alone has many specific properties and numerous applications, when combined with other materials, hybrid particles are formed that exhibit synergistic properties beyond the capabilities of the individual components [32]. GO hybrid particles are structures where graphene oxide is functionally combined with materials such as metal nanoparticles, metal oxides, biomolecules or organic polymers. This combination shows an improvement in mechanical, electrical and chemical properties and above all extends the application potential [32; 33].

The combination of GO with metallic nanoparticles such as silver, gold, copper leads to the formation of hybrid particles that exploit the conductivity of graphene oxide as well as the antibacterial, plasmonic or catalytic properties of metals [32]. GO-based metal oxide hybrids provide photocatalytic capabilities (GO-ZnO coupling), magnetic properties (GO-Fe₃O₄) and antibacterial and adsorption capabilities (GO-TiO₂). These combinations can most commonly be found in environmental technologies such as water purification, heavy metal removal or photodegradation of organic pollutants [32]. An important connection, especially in biomedical applications, is hybrid GO particles with biomolecules such as peptides, proteins, DNA, growth factors. They use the specific large surface area of GO to adsorb the biomolecules, functional groups in their structure for covalent binding of bioactive substances and also controlled release of substances. Applications for these hybrids include targeted delivery of growth hormones in tissue engineering, the use of smart scaffolds [33], immunomodulatory effects [34], biosensors and gene delivery systems. Finally, the hybridization of GO particles with polymers, whether natural or synthetic, enables the creation of biocompatible scaffolds, mechanically resistant membranes and controlled drug release systems [33].

Hybrid GO particles often exhibit significantly better properties compared to pure graphene oxide or single bonded substances. These synergistic properties are due to a combination of physical, chemical and biological interactions between GO and the bound materials. These improved properties include increased tensile strength, modulus of elasticity and resistance to cracking. These properties are created by the association of GO with polymers or inorganic oxides. Graphene oxide acts as a reinforcing material, which prevents the formation of micro-cracks in the structure and its presence also helps to improve the compactness and cohesion of the material [32; 35]. The ability of GO to form conductive pathways in a hybrid material due to its π -conjugated structure is most effective in

conjunction with conducting polymers or metal nanoparticles. Conductive hybrid materials find applications in electroactive tissues in tissue engineering, biosensors and energy storage [36]. Another important property of hybrid GO particles is biological activity and biointeractivity. Here, the adhesion and proliferation of cells – for example, osteoblasts - is promoted. Furthermore, myogenic, osteogenic and chondrogenic differentiation and last but not least the binding and transport of biomolecules. Due to these properties, GO hybrids find applications in regenerative medicine, wound healing, anti-inflammatory and anti-cancer therapies [33].

3.1 Application of GO hybrid particles

Thanks to the synergistic properties created by combining graphene oxide with the aforementioned materials, hybrid GO particles find applications in a wide range of modern technologies.

Especially in biomedicine and tissue engineering, where they are used as scaffolds for tissue regeneration, carriers for targeted delivery of drugs and growth factors, biosensors and antibacterial coatings [33]. Furthermore, in the field of energy, where hybrid GO particles are used to construct supercapacitors, fuel cells, anodes and cathodes in batteries. In this sector, they are characterized by their high cyclic stability, capacitive properties and high ion transfer rates [36]. In the environmental field they are applied in adsorption of heavy metals, photocatalytic degradation of organic pollutants, filtration and water purification [32]. It is also used in the industrial sector, where GO hybrids are used to enhance petroleum technologies [35], anti-corrosion coatings, smart textiles and self-cleaning surfaces. Hybrid particles increase material stability and durability, even under difficult physical and chemical conditions [32].

4 ATOM TRANSFER RADICAL POLYMERIZATION (ATRP)

Atom Transfer Radical Polymerization (ATRP) is one of the most important methods of Controlled Radical Polymerization (CRP), which enables the synthesis of polymers with well-defined molecular weights, narrow molecular weight distributions and complex architectures such as block, star or brush polymers [37].

Atom Transfer Radical Polymerization (ATRP) was first introduced in 1995 by a research team led by Krzysztof Matyjaszewski as an innovative approach to controlling radical polymerization with accuracy comparable to controlled anionic polymerization methods [37]. ATRP has enabled the efficient preparation not only of linear polymers but also of materials with complex structures whose synthesis was previously difficult or virtually impossible.

ATRP has rapidly gained popularity not only in the field of basic polymer research, but also in a number of applied disciplines such as smart materials development, tissue engineering, targeted drug delivery and surface modification [38]. Its significance lies in the fact that it provides a simple and versatile procedure that can polymerise a wide range of monomers under mild conditions with the possibility of precise control of the polymer structure.

4.1 ATRP mechanism

Atom Transfer Radical Polymerization (ATRP) is based on the reversible activation and deactivation of growth radicals through the transfer of a halogen atom between the polymer chain and the metal catalyst [37].

The polymerization reaction principle is explained in Figure 7. The reaction begins by the activation of a halogen initiator (halogen is labeled as X) in the presence of complex of metal in a low oxidation state and ligand (Mt^n/L), as is shown in Figure 7. The catalyst transfers the halogen atom X from the (macro)initiator P_n-X to metal complex, the metal increases its oxidation state ($X-Mt^{n+1}/L$), while an active radical P_n^* is generated. When it reacts with monomer M, the polymer chain grows. Since the balance of the reaction is significantly moved to inactive state, the halogen X from $X-Mt^{n+1}/L$ returns to growing chain P_n^* and the macroinitiator in sleeping state (P_n-X) is formed. As was mentioned, during polymerization, there is a constant equilibrium between the active radical and the dormant halogenated ends of the polymers. The balance is shifted to dormant state

($K_{\text{act}} \ll K_{\text{deact}}$) to ensure the controll of the reaction. [37; 39]. This equilibrium minimizes side reactions and provides precise control over the molecular weight, molecular weight distribution and structure of the resulting polymers.

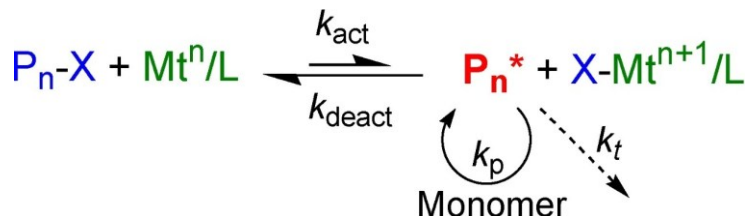


Figure 7: Mechanism of ATRP [40]

The ATRP reaction kinetics is determined by selection of reaction components and conditions, as is type and concentration of the metal catalyst, ligand, the choice of initiator, the type of monomer, solvent and the polymerization temperature.

4.2 ATRP components

The main components of the ATRP method are the catalytic system (transition metal halogens, ligands), initiators, monomers and solvents. The proper selection and concentration of the components is crucial set the kinetics of polymerisation, and the quality of the resulting polymer.

The catalytic system consists of a transition metal, most often copper, and a ligand that stabilizes the metal complex. The most commonly used ligands include bipyridine (bpy), tris(2-dimethylaminoethyl)amine (Me₆TREN), tris(2-pyridylmethyl)amine (TPMA), and PMDETA. The structure and stability of the metal–ligand complex directly influence the equilibrium constant (K_{ATRP}), thereby affecting the polymerization rate and control. The catalyst plays a key role in controlling the balance between active and dormant radicals, enabling precise control over the polymerization process. [37; 39; 41].

The choice of a suitable monomer is also crucial. It must be stable enough to avoid side reactions, but reactive enough to allow controlled polymerisation. The most commonly used vinyl monomers are acrylates (methyl methacrylate, butyl acrylate), acrylamides (*N*-isopropylacrylamide), styrene and its derivatives [39].

The initiator in an ATRP system is usually an alkyl halide ($R-X$), where X is most commonly bromine (Br) or chlorine (Cl). The most used initiators are ethyl-2-bromoisobutyrate (EBiB), methyl-2-bromopropionate (MBP) and benzyl bromide

derivatives. The choice of initiator determines the initial number of growing polymer chains and thus the resulting molecular weight of the polymer [37].

Finally, solvents are used to dissolve monomers and initiators, stabilize the catalytic system and control the polymerization kinetics. Commonly used solvents are toluene, anisole, acetonitrile (MeCN), dimethylformamide (DMF) and also the use of aqueous media for modified versions of ATRP. The choice of solvent has a major impact on the efficiency, speed and control of polymerization [38].

4.3 Types of ATRP

Since its initial introduction in 1995, various modifications have been made to the classic ATRP to remove some of its limitations, such as its high metal catalyst content or sensitivity to the presence of oxygen, while also seeking to expand its application possibilities. These modifications differ in the manner of catalyst initiation and regeneration. [37; 39].

4.3.1 Standard ATRP

This variant uses a halide initiator and a metal catalyst, most commonly copper and a ligand complex. A relatively high concentration of metal in the system is required and it is very sensitive to the presence of oxygen, which can deactivate the catalyst [37].

4.3.2 ARGET ATRP (Activators Regenerated by Electron Transfer)

This modification was developed to reduce the concentration of the metal catalyst. A reducing agent (e.g., ascorbic acid) is added to the system, which in the process regenerates the active Cu(I) from its oxidized Cu(II) form. The advantages of this modified method are lower toxicity, easier product purification and greater tolerance to the presence of oxygen [39].

4.3.3 ICAR ATRP (Initiators for Continuous Activator Regeneration ATRP)

This method has a similar principle to ARGET ATRP, but instead of a reducing agent, it uses a thermal initiator (e.g. AIBN) that generates free radicals and reduces Cu(II) to Cu(I). Both enable polymerization with very low metal catalyst content and higher oxygen tolerance [37].

4.3.4 AGET ATRP (Activators Generated by Electron Transfer)

Very similar to the previousARGET ATRP modification, but the reducing agent is added to the system during the preparation of the entire system. This method is advantageous for industrial applications and polymerization in aqueous media [37].

4.3.5 eATRP (Electrochemically Mediated ATRP)

The initiation and control of the polymerization takes place through the application of an electrical voltage that controls the redox equilibrium between the Cu(I) and Cu(II) complexes. The advantages of this method include very precise control of the polymerization and, above all, the possibility of starting and stopping the reaction by simply changing the voltage [39; 42].

4.3.6 photoATRP (Photochemically Mediated ATRP)

This method uses light activation to initiate the reduction of Cu(II) to Cu(I). It allows polymerization under mild conditions without the addition of a chemical reducing agent. The advantage is the temporal and spatial control of the polymerization by light [39; 42].

4.4 ATRP application

Atom transfer radical polymerization (ATRP) has become a key method for the preparation of functional polymers and nanostructured materials across many technological fields due to the high level of control over the polymerization process and the wide range of polymerizable monomers [38].

ATRP enables the synthesis of block copolymers, star polymers and brush polymers. These materials are used in areas where precise control of morphology, self-organization of polymers and formation of nanostructured surfaces are important [42]. Other uses are in biomedical applications in the preparation of scaffolds for tissue engineering, drug carriers, surface treatments of biomaterials [38]. This method is also used in the production of antifouling coatings, anti-corrosion protective coatings and functional sensing surfaces for the detection of biomolecules or environmental changes. These coatings prepared by ATRP allow precise control of the thickness, density and chemical function of the coatings [39]. More recently, ATRP has also been explored as a platform for controlled polymer depolymerization. Under appropriate conditions, polymers synthesized by ATRP can be

chemically recycled back to their monomers, offering new opportunities for sustainable materials and circular polymer lifecycles [43; 44; 45]

4.5 Advantages and limitations of ATRP

Atom transfer radical polymerization (ATRP) offers a number of advantages, but at the same time certain limitations of this method must be taken into account when designing the system [42].

The advantages of this method are precise control over molecular weight, where we control the controlled growth of polymer chains with predictable length and low polydispersity. Also the wide range of monomers that can be polymerized by this method. Furthermore, the possibility of synthesizing complex structures such as block copolymers, star, gradient and brush polymers. Another advantage is that the reaction can take place at relatively low temperatures and often in common organic solvents or even in water. A final advantage is the range of modified methods mentioned above, which bring extensions to the applications [39; 42].

However, there are several limitations. Even at low catalyst concentrations, traces of copper can remain in the product, which is a problem for biomedical and electronic applications. Therefore, metal catalysts and ligands must be removed after polymerisation, which can again complicate downstream processing. Classical ATRP is very sensitive to the presence of oxygen, which deactivates the catalyst. Another disadvantage is that the success of the polymerization is strongly influenced by the choice of ligand and not all ligand/metal combinations are suitable for all types of monomers [39; 42].

4.6 SI-ATRP

Surface-Initiated Atom Transfer Radical Polymerization (SI-ATRP) is a variant of ATRP that allows the growth of polymer chains directly from a solid substrate such as glass, metal, metal oxides or polymer films. This technique allows the formation of so-called polymer brushes, which are densely bonded polymer chains on a surface [46].

The main principle of SI-ATRP is the binding of the initiator to the surface of the substrate, from where the polymer chains grow gradually. In this way, the layer thickness, the density of the bonded brushes and the chemical composition of the surface can be controlled very precisely [47]. This method is also very sensitive to oxygen, but various

modifications such as SI-ARGET ATRP, photoinduced SI-ATRP or SI-ZnO-ATRP have also been developed that use metal platelets as sources of active species [48].

The application of polymer brushes finds use in areas of sensor manufacturing where polymer layers can be targeted to be sensitive to solvents and gases [47]. Furthermore, in antifouling and biomedical coatings that are resistant to protein and cell deposition, which is suitable, for example, for the manufacture of implants [46; 47]. Also, this method is used in nanotechnology to modify nanoparticles [46].

The advantages of SI-ATRP include high control over surface properties – hydrophobicity, bioactivity, sensitivity to stimuli, the ability to work in aqueous environments and the ability to form complex structures of the resulting polymers [46].

The polymerization method used in this work is surface-initiated ATRP, which allows precise control over polymer growth from the surface of GO. This method was chosen for its ability to produce well-defined polymer brushes and for its compatibility with the functionalization strategy used in this study.

5 MEASUREMENT METHODS

Several analytical techniques were employed in this thesis to monitor the course of polymerization, confirm the structure of the synthesized polymers, and characterize the resulting hybrid materials. Each method offers specific insights – from molecular structure and composition (NMR), to molecular weight distribution (GPC), chemical functionality (FTIR), thermal stability (TGA), and electrical properties. The following subsections briefly describe the principles of each technique and their application in this research.

5.1 Nuclear Magnetic Resonance Spectroscopy (NMR)

Nuclear Magnetic Resonance (NMR) spectroscopy is a powerful analytical technique used to determine the molecular structure, composition, and dynamics of organic compounds, including polymers. It is based on the absorption of radiofrequency radiation by atomic nuclei in a strong magnetic field. Among the most commonly observed nuclei in polymer chemistry are hydrogen (^1H) and carbon (^{13}C), whose magnetic properties enable precise identification of different chemical environments within the polymer chain. In high-resolution NMR, each chemically non-equivalent nucleus produces a distinct signal whose position (chemical shift, δ) depends on the electronic environment surrounding the atom. This allows the identification of monomer units, end-groups, and even stereoregularity (tacticity) in polymers. For example, in poly(glycidyl methacrylate), the ^1H NMR spectrum reveals signals corresponding to the glycidyl and methacrylate units, providing evidence of successful polymerization and the structure of the side chains.

In the context of this thesis, ^1H NMR was used to verify the composition and conversion of glycidyl methacrylate (GMA) during atom transfer radical polymerization (ATRP) reactions. By integrating selected peaks corresponding to monomer and polymer regions, the monomer conversion could be calculated. Additionally, signal broadening was observed with increasing molecular weight, which is a typical phenomenon due to restricted mobility of polymer chains.

NMR remains one of the most versatile tools in polymer science. It provides detailed, non-destructive information on polymer composition, chain architecture, and end-group functionality, which is essential for both academic research and industrial applications [49; 50].

5.2 Gel permeation chromatography (GPC)

Gel permeation chromatography (GPC) is a basic analytical method for determining the molecular weight of polymers and their distribution. The principle of GPC is based on separating molecules according to their hydrodynamic volume as they pass through a porous column. Larger molecules are excluded from the porous material and elute earlier, while smaller ones penetrate deeper into the pores and elute later [51].

The technique was first described by Moore in 1964, who proposed the use of porous gel phases for the characterization of polymer systems. Since then, GPC has become the standard method for characterizing synthetic and natural polymers and is widely used in both academic and industrial settings. GPC provides not only average molecular weights (number average molecular weight and weight average molecular weight, M_n and M_w , respectively), but also a dispersion index ($\mathbb{D}$), which indicates the degree of uniformity of the distribution of polymer chain masses [52].

The accuracy of the measurement depends on the calibration of the system - polystyrene-based standards are most commonly used, as their behavior is well described. For polymers with different structures (e.g., with branching, brush segments, etc.), the hydrodynamic behavior may differ, and possible systematic deviations must therefore be taken into account. An alternative is absolute measurement using detectors based on light scattering (LS) or viscosity detectors [51; 52].

In this work, gel permeation chromatography (GPC) was a key tool for the quantitative characterization of polymer products. Using this technique, the experimental M_n was determined, which was then compared with the theoretically calculated value based on monomer conversion determined by ^1H NMR. Furthermore, the $\mathbb{D}$ was monitored as an indicator of controlled way if reaction. GPC also provided information on the distribution of molecular weights through elution curves and allowed the kinetics of polymerization to be monitored based on time-resolved samples. This data was essential for designing of polymerization conditions on the efficiency and controllability of polymer brush synthesis in the GO/PGMA system.

Thanks to its ability to provide quantitative and repeatable data on the molecular weight of polymers, GPC is one of the most important techniques for characterization of polymer materials.

5.3 Fourier transform infrared spectroscopy (FTIR)

Fourier transform infrared spectroscopy (FTIR) is a widely used analytical technique for the qualitative and quantitative identification of functional groups in organic and inorganic compounds. It is based on the principle of absorption of infrared (IR) radiation by molecules, which then transition to vibrationally excited states. Each type of chemical bond absorbs IR radiation at a characteristic wavelength, creating a spectral "fingerprint" specific to that substance. Infrared radiation is filtered and analyzed using Fourier transformation, which converts the measured interferograms into an absorbance or transmittance spectrum depending on the wave number. The spectrum can be divided into several regions – e.g., the valence vibration region (4000–1500 cm^{-1}), where typical bands for –OH, –NH, –CH, or =C=O groups are found, and the fingerprint region below 1500 cm^{-1} , which is specific to the overall structure of the molecule [53].

Thanks to its speed, simplicity, and non-invasiveness, FTIR is ideal for characterizing polymer materials, monitoring chemical reactions, and verifying the structure of synthesized products. In polymer chemistry, FTIR is used to monitor the presence and changes of functional groups during polymerization or modification of polymer chains. For example, in glycidyl methacrylate-based polymers (PGMA), FTIR can be used to monitor the disappearance of vinyl groups (C=C) during polymerization, as well as the presence of epoxy and ester groups [53; 54].

In this work, FTIR spectroscopy was used to confirm the presence of characteristic functional groups in GO-g-PGMA hybrid particles. The spectra were used to verify the success of grafting of the polymers on the particle surface.

5.4 Thermogravimetric analysis (TGA)

Thermogravimetric analysis (TGA) is a thermal analytical technique that allows the monitoring of changes in the weight of a sample as a function of temperature or time under controlled environmental conditions. In polymer chemistry, TGA is an indispensable tool for evaluating thermal stability, determining the content of fillers and additives, and analyzing degradation processes in polymer materials [55].

The principle of TGA is based on measuring the weight changes of a sample during heating, cooling, or maintenance at a constant temperature. The sample is placed on a highly sensitive balance inside a furnace, where it is exposed to a precisely controlled temperature

program. During the analysis, various physical and chemical processes may occur, such as evaporation, decomposition, oxidation, or desorption, which are reflected in changes in the sample's weight. The result of the measurement is a thermogram, which shows the dependence of weight on temperature or time [55; 56].

In polymer analysis, TGA is used to determine the decomposition temperature of a polymer, its moisture content, volatile substances, fillers, and residual ash. This data is crucial for understanding the thermal behavior of polymer materials and their suitability for specific applications. For example, TGA analysis can reveal the temperature range in which a polymer is stable, which is important for its processing and final use [55; 56].

In this work, TGA was used to evaluate the amount of polymer grafted on the particles surface. This information is essential for estimation of grafting density of the polymer brushes. Analysis of the thermograms allowed the identification of temperatures at which significant weight losses occur, providing information on the stability of the polymer chains.

5.5 Electrical conductivity

Electrical conductivity is a key parameter in characterizing conductive polymers and their composites, especially in applications such as sensors, antistatic materials, electromagnetic shielding, and flexible electronics. Since most polymers are electrically non-conductive under normal conditions, conductivity is usually achieved by adding conductive fillers (e.g., carbon nanotubes, graphene) or by chemically doping conjugated polymers [57].

Several methods are most commonly used to measure the electrical conductivity of polymer materials. The two-point method is a simple technique where the resistance between two electrodes connected to the sample is measured. The disadvantage is the influence of contact resistance, which can distort the results, especially in materials with low conductivity. This method is suitable for less conductive samples up to 10^{-4} S/cm. The four-point method is also used, which is a more accurate method that eliminates the influence of contact resistance. The current is conducted through external electrodes, while the voltage is measured between the internal electrodes. This method is, on the other hand, suitable for more conductive samples from 10^{-4} S/cm. Last but not least, impedance spectroscopy is also used, which allows the analysis of the frequency dependence of the electrical properties of a material, which is useful for studying dielectric properties and ionic conductivity [57; 58].

One of the key aspects studied in this thesis is the change in electrical conductivity of GO after polymer grafting. It was revealed, that tertiary amines with short aliphatic substituents, including PMDETA, can reduce the GO surface during SI-ATRP [59]. Therefore the ATRP reaction conditions were selected to reach this effect. The values of electrical conductivity of GO-g-PGMA did not exceed the 10^{-4} S/cm value, therefore the two-point method was used in this study.

II PRACTICAL PART

6 AIM OF THE WORK

The present thesis is focused on the preparation of graphene oxide grafted with poly(glycidyl methacrylate) GO-g-PGMA using surface-initiated atom transfer radical polymerization (SI-ATRP). Before the polymer grafting step, homopolymerization of GMA was studied and optimized to ensure good control over the reaction and minimize undesirable crosslinking. Various analytical techniques were employed to evaluate both the polymerization process and the properties of the resulting materials.

The main aims of the thesis are:

- Optimization the reaction conditions for the ATRP homopolymerization of GMA, with a focus on controlling molecular weight and reducing oxirane group crosslinking.
- Synthesis GO-g-PGMA hybrid particles via SI-ATRP under optimized conditions.
- Characterization of the resulting polymers and hybrid materials using ^1H NMR, GPC, FTIR, TGA, and electrical conductivity measurements.
- Evaluate the effect of polymerization conditions on the electrical conductivity of the GO-g-PGMA particles.

7 CHEMICALS AND EQUIPMENT

7.1 Chemicals

Monomer glycidyl methacrylate (GMA), initiator ethyl 2-bromoisobutyrate (EBiB), ATRP ligand *N,N,N',N'',N'''*-pentamethyldiethylenetriamin (PMDETA), CuBr, CuBr₂, anisole, chloroform, dried tetrahydrofuran (THF), *d*-chloroform, basic and neutral aluminium oxide. These chemicals were purchased from Sigma Aldrich.. The monomer was passed through basic alumina column before use. All other chemicals were used as received. The GO particles modified with ATRP initiator 2-bromoisobutyryl bromide (GO-I) was prepared according to literature [59] and it was supplied by supervisor.

7.2 Methods

The organic fraction content in the GO-g-PGMA hybrids was calculated based on thermogravimetric analysis (TGA), using TA instruments Q50 thermogravimetric analyzer (New Castle, Delaware, USA) FROM 30 TO 800 °C with a heating rate of 5 °C min⁻¹ under nitrogen flow 15 ml min⁻¹. Monomer conversion was calculated based on proton nuclear magnetic resonance (¹H NMR) JEOL ECZ400R/S3 Spectrometer (JEOL, USA). Deuterated chloroform was used as NMR solvents. Molecular parameters of polymers: numbers of average molar mass (*M_n*) and dispersity (*Đ*) were determined using gel permeation chromatography (GPC) Waters HPLC e2695 s equipped with RI detector 2414 and columns: PL gel MIXED-A (300 x 7,5 mm, 20 μm), PL gel MIXED-B (300 x 7,5 mm, 10 μm) and PL gel MIXED-D (300 x 7,5 mm, 5 μm). THF was used as eluent; the flow rate equaled 1 mL min⁻¹ and the measurement temperature was 40 °C. PS standards were used for calibration. To confirm polymer grafting on the GO surface, Fourier transform infrared (FTIR) spectroscopy was applied using a Nicolet iS5 FTIR spectrophotometer (Thermo Fisher Scientific, Inc., USA) equipped with an attenuated total reflectance (ATR) mode and germanium crystal. Measurements were performed in the range of 4,000–600 cm⁻¹ using 64 scans. The electrical conductivity of GO and GO-g-PGMA samples was characterized by the two-point method. Before the measurement, the pellets with a diameter of 13 mm and thickness of 2.2 – 2.4 mm were prepared by compressing at the hydraulic pressure of 7 MPa. The samples were characterized by the two-point method: The pellets placed between electrodes and measured using Keithley 2400 SourceMeter (Keithley, USA).

8 OPTIMISATION OF REACTION CONDITIONS

The first stage of the whole work was to optimize the GMA homopolymerization reaction conditions. The selected conditions were then utilized for grafting of polymers from the particle surface. Totally, nine reactions were performed, six homopolymerizations and three polymerizations from particles surface.

The initial reaction conditions were addapted from literature [60], where reduction of GO during grafting of styerene was performed. In the Table 1 the calculation of the amount of monomer (M), initiator (I), ligand (L), catalytic system Mt^n and Mt^{n+1} , and solvent (S) is shown.

Table 1: Amount of chemicals in the reactions

	GMA	EBIB	PMDETA	CuBr	CuBr ₂	anisoole
	M	I	L	Mt^n	Mt^{n+1}	S
ratio	200	1	1	0,4	0,1	50 %
n [$\times 10^{-4}$ mol]	377,0	1,880	1,880	0,753	0,188	
M [g/mol]	142,15	195,05	173,3	143,45	223,35	
m [g]	5,350	0,037	0,033	0,011	0,004	
ρ [g/cm ³]	1,07	1,33	0,83			
V [ml]	5,000	0,028	0,039			5,000

8.1 Procedure to optimise reaction conditions

In the first stage of the procedure, the monomer was purified by passing through aluminium oxide column and bubbled with argon for 30 minutes. The solvent anisoole was bubbled 30 minutes as well. Then, solid components – CuBr, CuBr₂ were put into the Schlenk flask. Afterwards, the air was evacuated from the flask and the space was refilled with Ar. This procedure was repeated three times. The Anisoole (3 mL) and GMA were added to reaction flask under argon flow. The stock solution of PMDETA (54 μ l in 2mL anisoole) was prepared, purged with argond and 1mL solution was added to reaction flask. Similarly, the sacrificial initiator EBIB was added to reaction flask. The flask was immersed into the oil bath heated to the desired temperature and the reaction was started. . The 0,1 mL samples were taken from the reaction in hourly intervals to obtain data for kinetics evaluation.

8.2 Sample collection and and preparation for measurement

Samples were collected at intervals into small glass vials. Two samples were taken at any given time, one for subsequent analysis by proton nuclear magnetic resonance (¹HNMR) spectroscopy and the other for gel permeation chromatography (GPC).

Samples for ^1H NMR were prepared with deuterated chloroform. First, a sample with a small amount of d-chloroform was purified through alumina and collected in a new vial. Then a small amount of d-chloroform was added again. Finally, 0.6 ml was taken into a glass cuvette and the sample was given for measurement.

Sample preparation for gel permeation chromatography measurements was similar, but a different solvent was used. First, the sample was mixed with a small amount of tetrahydrofuran (THF), purified through alumina to remove copper and then diluted again with THF and the sample was submitted in a vial for measurement.

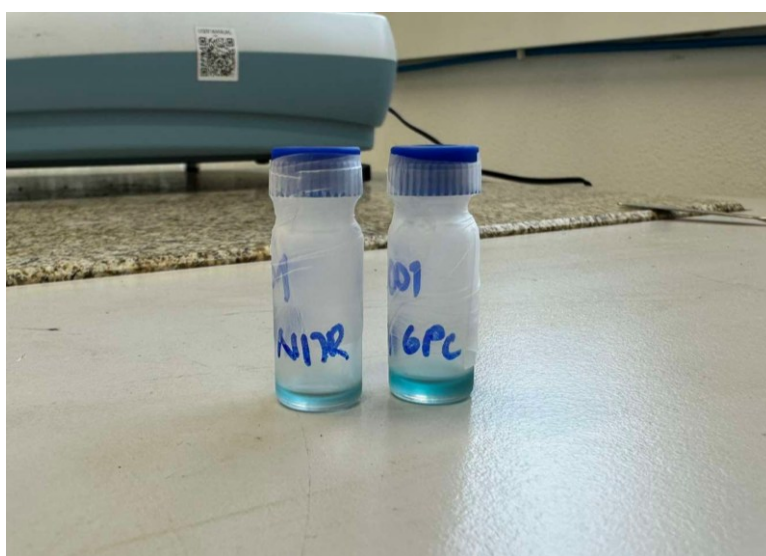


Figure 8: Samples collected in vials

9 RESULTS AND DISCUSSION

The homopolymerization of GMA was performed according to conditions summarized in the Table 2 entry 1-7.

Table 2: the ATRP reaction conditions of GMA

Reaction No.	M:I:L:CuBr:CuBr ₂ :S	time [h]	T [°C]
1	200:1:1:0,4:0,1:0,5	5	80
2	200:1:1:0,4:0,1:0,5	44	30
3	200:1:1:0,4:0,1:0,5	94	45
4	200:1:1:0,4:0,1:0,5	91	40
5	200:1:1:0,4:0,1:0,5	238	30
6	200:1:1:0,4:0,1:0,5	-	30
7	200:1:1:0,4:0,1:0,5	26,5	30

Optimization of ATRP conditions

According to the literature [60] the catalytic system CuBr/PMDETA was chosen and the reaction temperature was set to 80°C. At these conditions the polystyrene (PS) was grafted from the surface and GO was reduced. To obtain the increase of electrical conductivity upto 8 orders, the reaction is supposed to run at least four days.

The conditions listed in Table 2, entry 1 were utilized in the reaction No1. After one hour the polymer with M_n 7,400 g/mol and D 1.38 was obtained. This result showed poor control of the reaction. Moreover after 5 hours the gel was formed. The high temperature initiated the crosslinking reaction of oxirane pendant groups. Thus in the reaction No. 2 the temperature was decrease to 30 °C. These conditions enabled to run the reaction significantly longer time (44 hours). The kinetics was analyzed and the results are summarized in Table 3.

Table 3: Results of polymerization No.2: Number average molecular weight M_n , weight average molecular weight M_w , dispersion D , monomer conversion, theoretical molecular weight $M_{n\text{ theor}}$

Reaction time [hours]	M_n [g/mol]	M_w [g/mol]	D	Conversion [%]	$M_{n\text{ theor}}$ [g/mol]
0,5	5400	6500	1,21	2	600
1	5200	6600	1,26	6	1700
2	5400	6900	1,28	10	2800
22	6000	8600	1,43	13	3700
44	6300	8600	1,36	17	4800

The GPC results showed that similar molecular weight was observed within first two hours of the reaction, the M_n slightly risen after the longer reaction time, but it also lead to loss of controll. It can also be clearly seen on GPC traces depicted in the Figure 9. The broadening of the MW distribution after 22 hours is clearly visible.

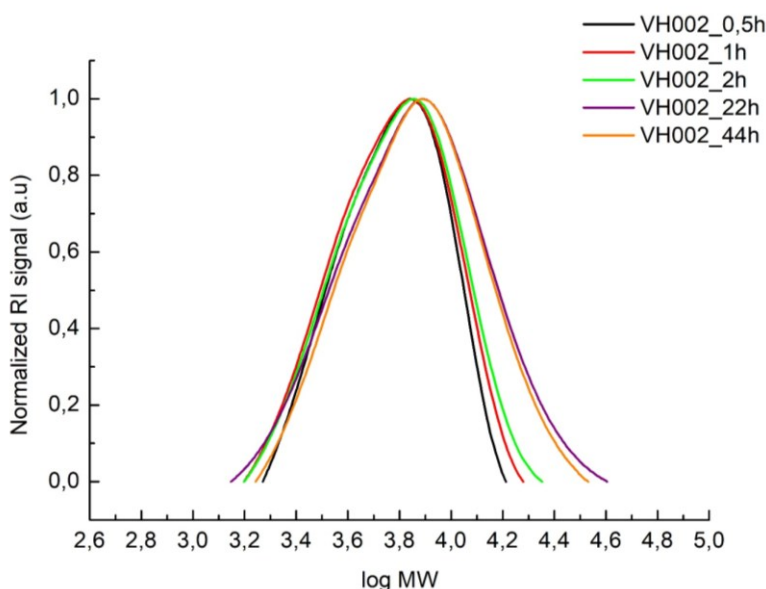


Figure 9: GPC traces of reaction No.2

These results suggest that although gelation was suppressed, the reaction conditions still require improvement to increase both conversion and molecular weight distribution.

Paralelly to reaction No. 2 the reaction No.3 and reaction No.4 were running at 45 °C and 40°C, respectively. These conditions were studied to find the limiting temperature for gelation. The results are summarized in Table 4 and 5. These condition enabled to extend the reaction time to ~ 90 hours.

Table 4: Results of reaction No.3: Number average molecular weight M_n , weight average molecular weight M_w , dispersion $\mathcal{D}$, monomer conversion, theoretical molecular weight $M_{n\text{ theor}}$

Reaction time [hours]	M_n [g/mol]	M_w [g/mol]	$\mathcal{D}$	Conversion [%]	$M_{n\text{ theor}}$ [g/mol]	Note
0,5	4900	6300	1,29	6	1700	
1	5300	6700	1,26	9	2600	
2	5700	7400	1,29	11	3100	
22	5800	7700	1,32	20	5700	
94	NA	NA	NA	NA	NA	gel

Table 5: Results of reaction No.4: Number average molecular weight M_n , weight average molecular weight M_w , dispersion $\mathcal{D}$, monomer conversion, theoretical molecular weight $M_{n\text{ theor}}$

Reaction time [hours]	M_n [g/mol]	M_w [g/mol]	$\mathcal{D}$	Conversion [%]	$M_{n\text{ theor}}$ [g/mol]	Note
0,5	7400	9500	1,28	8	2300	
1	8100	10 800	1,33	16	4500	
1,5	7900	11 100	1,4	19	5400	
2	7700	10 900	1,42	22	6300	
22	7500	10 400	1,38	27	7700	
43	7500	10 400	1,39	30	8500	
69	7900	10 600	1,34	32	9100	
91	NA	NA	NA	NA	NA	gel

GPC traces of reaction No.3 are depicted in Figure 10. The evolution of molecular weight in time was obtained. However the bimodality of the peaks in lower MW is visible, even after 30 min. Similar feature can be seen also in reaction No. 2. The MW is also higher then theoretical MW. The reason of higher MW then predicted is usually associated with less efficient initiation. The less chains with higher MW then expected is created. On the other hand, the bimodality of the peak may also reffer to the presence of oligomers in monomer batch.

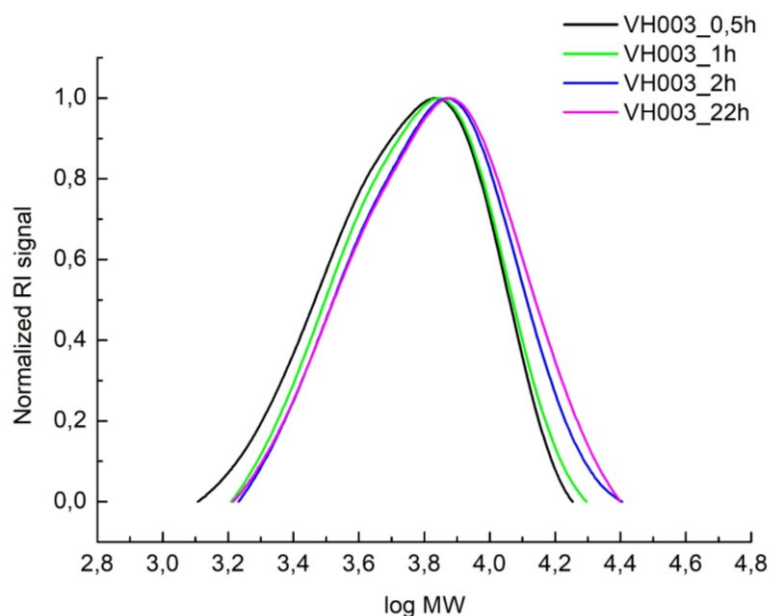


Figure 10: GPC traces of reaction No.3

In the reaction No. 5 another batch monomer was used. The temperature 30°C was selected. The results are summarized in the Table 6.

Table 6: Results of reaction No.5: Number average molecular weight M_n , weight average molecular weight M_w , dispersion $\mathcal{D}$, monomer conversion, theoretical molecular weight $M_{n\text{ theor}}$

Reaction time [hours]	M_n [g/mol]	M_w [g/mol]	$\mathcal{D}$	Conversion [%]	$M_{n\text{ theor}}$ [g/mol]
0,5	4900	5600	1,14	4	1100
1	5400	6100	1,13	5	1400
1,5	5100	5900	1,16	6	1700
2	4800	5700	1,19	7	2000
22	6000	8900	1,48	15	4300
46	5700	8500	1,49	20	5700
70	5900	8200	1,39	22	6300
215	6000	8300	1,38	25	7100
238	5800	8300	1,43	27	7700

The first two hours proceeded satisfactorily, the MW values obtained by GPC fits the predicted MW values. After 22hours the broadening of the MW distribution was observed. Therefore it was concluded that GMA can be polymerized only at mild temperatures and short reaction times (few hours) to keep controll. The reaction conditions No.5 were adapted for SI-ATRP. To verify that conclusion, the reaction was repeated as No.6. Unfortunately, the reaction was not running, thus reaction No.7 was performed.

This reaction No.7 was carried out at a temperature of 30 °C for a total reaction time of 26.5 hours. After 1,5 hours, the monomer conversion 6 % was obtained, which is comparable value to 7 % obtained in reaction No.5. Similarly, the theoretical values of MW do not meet the data obtained from GPC. The reason can be also related to the calibration limit of the GPC columns. To conclude the optimization studies, the reaction conditions No.5 were selected as suitable for SI-ATRP from GO particles

Table 7: Results of reaction No.7: Number average molecular weight M_n , weight average molecular weight M_w , dispersion D , monomer conversion, theoretical molecular weight $M_{n\text{ theor}}$

Reaction time [hours]	M_n [g/mol]	M_w [g/mol]	D	Conversion [%]	$M_{n\text{ theor}}$ [g/mol]
0,5	5200	5900	1,13	2	600
1	4500	5100	1,13	4	1100
1,5	4200	5100	1,22	7	2000
2	NA	NA	NA	NA	NA
26,5	5500	8400	1,53	12	3400

9.1 Preparation of GO-g-PGMA hybrid particles

To prepare hybrid GO-g-PGMA particles (reaction No.8 and No.9), SI-ATRP of GMA from GO-I particles was performed. The conditions of reaction No.5 were slightly modified. The concentration of ligand was increased. This strategy is the another option how to increase the electrical conductivity of the GO particles, as high temperature and long reaction time cannot be used due to gelation. Reaction No. 9 is the upscale of reaction No.8.

The grafting of GMA from GO was performed according to conditions listed in Table 2. The 100 mg of GO modified with ATRP initiator bromoisobutyl bromide were added to reaction. These particles were received in the research group and the modification of GO with initiator was performed according to literature [59]. Polymerization was carried out at 30°C for 7 h. Monomer conversion reached 25 % and M_n was 6600 g/mol with a D of 1,3. These characterizations were performed at polymers grown from sacrificial initiator.

The upscale reaction No.9 was performed at 30 °C, but for a shorter period of 4 hours. The reaction conditions are listed in Table 9. The monomer conversion was not evaluated in this reaction. M_n was 6400 g/mol with a D of 1,3.

Table 8: Amount of chemicals in the reaction No.9

	GMA	EBIB	PMDETA	CuBr	CuBr ₂	Anisole	GO-I [g]
ratio	200	1	4	0,4	0,1	50 %	
n [$\times 10^{-4}$ mol]	377,0	1,880	1,880	0,753	0,188		
M [g/mol]	142,15	195,05	173,3	143,45	223,35		
m [g]	5,35	0,0367	0,1304	0,108	0,042		1,000
ρ [g/cm ³]	1,0700	1,3290	0,8300				
V [ml]	50,000	0,276	1,572			50,000	

The particles GO-g-PGMA were purified from the reaction mixture by centrifugation and filtration. The reaction mixture was diluted with tetrahydrofuran and acetone in a ratio of 1:1 and centrifuged at 4000 rpm for 10 minutes. The GO-g-PGMA were collected, dispersed in chloroform, sonicated in ultrasonic bath and filtered over 0.44 microns PTFE filters. The particles on the filter were washed with chloroform and acetone. The filtration procedure was repeated three times. The purified GO-g-PGMA was placed to vacuum oven at dried at 30°C and 10 mbar for three days. The dried samples were characterized by FTIR, TGA and electrical conductivity.

9.2 Fourier-transform infrared spectroscopy (FTIR)

Fourier transform infrared spectroscopy (FTIR) was used to monitor functional groups present in GO, GO-I, and GO-g-PGMA samples, thereby confirming the course of chemical modifications. The spectra allow comparison of changes in characteristic absorption bands corresponding to individual chemical bonds during the various stages of synthesis – from the initial graphene oxide (GO) through the initiated surface (GO-I) to the final hybrid particles with bound polymer brushes (GO-g-PGMA).

Based on the occurrence or attenuation of certain peaks, it is possible to confirm, for example, the presence of epoxy or ester groups and simultaneously monitor their transformation during polymerization. The recorded changes in the GO-g-PGMA spectrum compared to the initial materials indicate successful polymerization of GMA on the GO surface and the formation of hybrid structures. The following graph shows a comparison of the spectra of all three samples and serves as a basis for detailed interpretation of the individual functional groups.

The FTIR spectra are shown in the Figure 11. The stretching of C=O group can be visible at 1650 cm⁻¹. The GO-g-PGMA sample shows broadening of this band to higher

wavenumber, highlighted by arrow in the graph. This feature can be attributed to carbonyl groups present in polymer structure. The presence of polymer on the GO surface is further confirmed by novel sharp peak at $1,140\text{ cm}^{-1}$ that corresponds to the scissoring of the $-\text{CH}_2$. The oxirane group is visible at 830 cm^{-1} , after modification this band increased the intensity significantly.

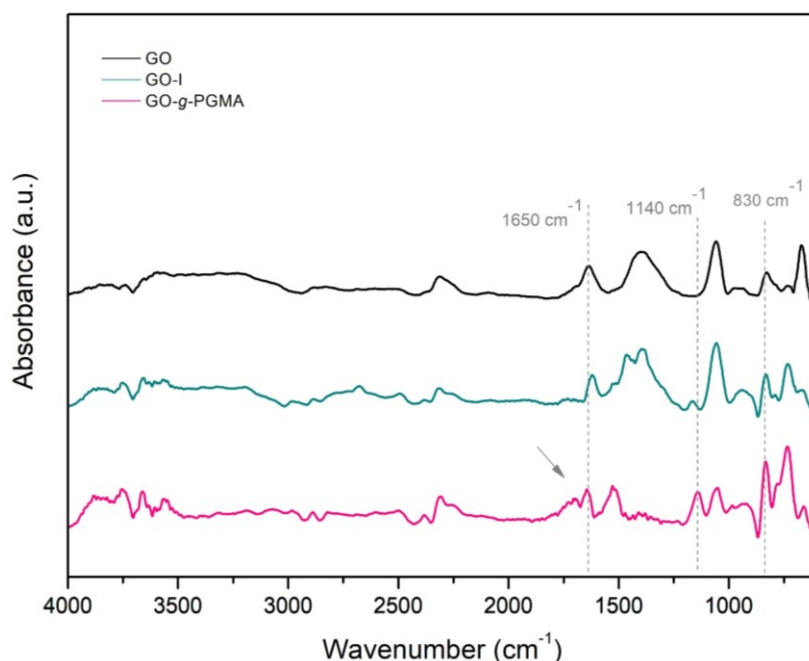


Figure 11: FTIR spectra of neat GO, GO modified with initiator (GO-I) and GO grafted with PGMA brushes (GO-g-PGMA)

9.3 Thermogravimetric analysis (TGA)

Based on theoretical knowledge, thermogravimetric analysis (TGA) was used to verify the presence of polymer brushes bound to the surface of graphene oxide and to assess their quantity. The measurements were performed on GO and GO-g-PGMA samples under defined conditions (in an inert atmosphere, heated to $800\text{ }^{\circ}\text{C}$). The results are shown in the Figure 12. By comparing the thermograms of both samples, it was possible to identify specific temperature ranges in which the individual components decompose and to quantify the proportion of grafted polymer. This information was then used to evaluate the success of the polymerization and the effectiveness of the particle surface modification.

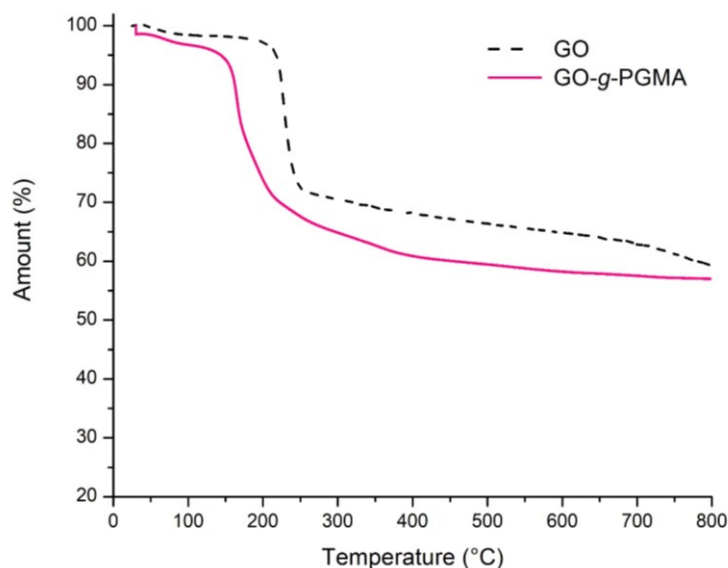


Figure 12: TGA curves of the GO and GO-g-PGMA

Spectra of both samples show an initial weight loss in the range of 100–150 °C due to the evaporation of bound water. Another weight drop occurs between 220–260 °C and it corresponds to the decomposition of oxygen containing functional groups present on the GO surface including (-COOH and -OH). The spectra of GO-g-PGMA sample are shifted to lower temperatures due to lower stability of the functional groups in presence of longer substituents. The weight loss at this temperature range was 28 wt % and 27 wt % for GO and GO-g-PGMA samples, respectively. The GO-g-PGMA sample exhibits another weight loss drop between 200–400 °C, which corresponds to the decomposition of grafted PGMA chains. The amount of polymer was estimated to 6 wt %, which is similar to the value obtained in previous study [61]. The similar content of oxygen containing groups in both samples confirmed the poor reduction of GO obtained during grafting proces.

9.4 Electrical conductivity

The electrical conductivity of the prepared samples in tablet form was determined using the two-point method. This method was chosen because samples exhibited electrical conductivity lower than 10^{-4} S.cm⁻¹. The aim of the measurement was to determine the influence of polymerization and the structure of polymer chains on the resulting electrical properties of the samples.

The electrical conductivity of the pristine GO was measured to be $(6.0 \pm 0.3) \times 10^{-8}$ S/cm. A slight decrease in conductivity was observed for GO-I

$(3.1 \pm 0.5) \times 10^{-8}$ S/cm, which can be attributed to partial blocking of the conductive pathways by the initiator molecules [61]. After the surface-initiated polymerization, the conductivity of the GO-g-PGMA sample significantly increased to $(5.4 \pm 0.2) \times 10^{-7}$ S/cm. This enhancement can be ascribed to the partial reduction of GO that occurs during the polymer grafting process, which restores some of the sp^2 hybridized carbon network, thus improving the electron transport properties. . Similar results were obtained in literature [61]. The results are summarized in Table9.

Table 9: Electrical conductivity of GO, GO-I and GO-g-PGMA particles

Sample	Electrical conductivity σ [S/cm]
GO	$(6.0 \pm 0.3) \times 10^{-8}$
GO-I	$(3.1 \pm 0.5) \times 10^{-8}$
GO-g-PGMA	$(5.4 \pm 0.2) \times 10^{-7}$

CONCLUSION

This thesis focused on the synthesis of hybrid particles based on graphene oxide grafted with poly(glycidyl methacrylate) brushes via surface-initiated atom transfer radical polymerization. The experimental work was carried out in two main phases.

In the first phase, it was crucial to optimize the polymerization process. This was supported by kinetic analysis of reactions No. 1–7, which revealed that using a fresh GMA monomer and conducting the polymerization at 30 °C led to high monomer conversion and rapid increase in molecular weight. These results indicated efficient polymer growth within a short reaction time.

In the second phase, GO-g-PGMA hybrid particles were prepared under the optimized conditions..

The FTIR proved the presence of PGMA on the GO surface. The TGA showed the polymer content on the GO-g-PGMA sample reached 6 wt %. The electrical conductivity measurements showed that the reaction conditions applied for grafting of PGMA onto GO resulted in a slight increase in electrical conductivity. The oxirane pendant group did not allow to use higher temperature than 30°C and longer reaction time then 4 hours to keep control way of reaction.

Overall, the study demonstrates the feasibility of using SI-ATRP to prepare functional GO-based hybrid materials with tunable surface and electronic properties. These materials have potential applications in fields such as sensing, electronics, or biomedical engineering.

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LIST OF ABBREVIATIONS

GO	Graphene oxide
rGO	Reduced graphene oxide
GMA	Glycidyl methacrylate
PGMA	Poly(glycidyl methacrylate)
ATRP	Atom Transfer Radical Polymerization
SI-ATRP	Surface-Initiated Atom Transfer Radical Polymerization
NMR	Nuclear Magnetic Resonance
GPC	Gel Permeation Chromatography
FTIR	Fourier-Transform Infrared Spectroscopy
TGA	Thermogravimetric Analysis
PMDETA	N,N,N',N',N''-Pentamethyldiethylenetriamine
bpy	2,2'-Bipyridine
TPMA	Tris(2-pyridylmethyl)amine
Me ₆ TREN	Tris(2-(dimethylamino)ethyl)amine
M _n	Number-average molecular weight
M _w	Weight-average molecular weight
Đ	Dispersity (M _w /M _n)
VEGF	Vascular endothelial growth factor
MW	Molecular weight