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Applications of Xanthate Metal Compounds in the Last Ten Years: A Review

Aplicações de Compostos Metálicos de Xantatos nos Últimos Dez Anos: Uma Revisão

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Xanthates are organic compounds that have high versatility and low production cost. These factors make xanthates promising in several areas of research, such as environmental, biological and technological. In the environmental field, xanthates are widely used to remove heavy metals from wastewater. In addition, due to the presence of sulfur and oxygen atoms in their structure, they can coordinate themselves to metal centers in different ways, forming coordination compounds. These compounds have been investigated mainly in the biological area, in which several studies of their anticancer and antibacterial properties have been reported. In the technological area, the formation of metal sulfides by thermal decomposition of the xanthate complexes has been of great interest for obtaining thin films and solar cells. In this review, applications of xanthates and their coordination compounds to these areas, are reported over the past ten years.

Keywords: Xanthates; heavy metal; flotation; cellulose; anticancer; antibacterial; thin films.

1. Introduction

The xanthates (dithiocarbonates) are a class of organic compounds containing the characteristic group -OCS_2 (Figure 1), and are classified as an ester or a salt of xanthic acid.[1]

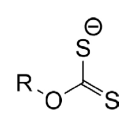
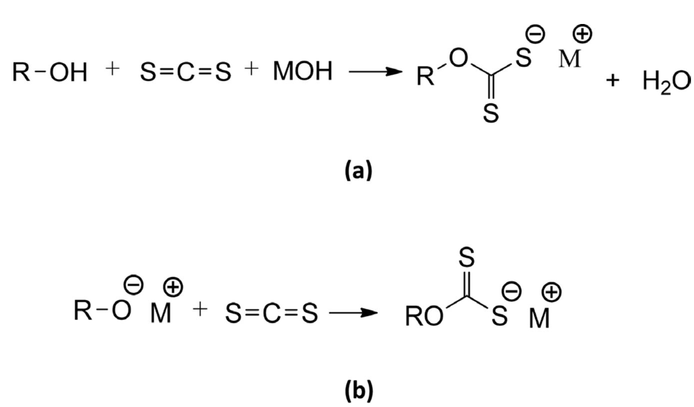


Figure 1. General structure of xanthate anion

Research involving xanthates dates back to 1834 when the first xanthate was discovered by Zeise.[2] The Greek term "xanthos" gave rise to the name of this class because it means the yellow color, which is characteristic of these compounds. During a period, xanthates were used only in laboratories, but later some derivatives started to be used as fungicides and in stages of the rubber manufacturing process. In 1923, a study by Cornelius Keller indicated the use of xanthates as collectors for flotation of heavy metal sulfide minerals. Thus, xanthate has become the first water-soluble organic compound to be used as a collector. Over the years, xanthate research has grown and continues to expand across diverse areas of research and applications.[3] Xanthates can be obtained through simple processes, as shown in Scheme 1.[4]



Scheme 1. Synthesis of xanthate salts from: **(a)** an alcohol and disulfide[4] **(b)** an alkoxide[4]

This characteristic is justified by the ability of these atoms to form bonds with metals through the free electrons present in their p-type orbitals.[5,6] Due to their wide variety of coordination modes (Figure 2), the most promising application of these compounds has been their use as flotation agents. Many studies have reported xanthate use to remove heavy metals from wastewaters, mainly because of the low economic value when compared to other flotation agents. The definition of heavy metals is not precise; however, it is commonly defined as elements that have a high atomic weight and a density five times higher than water.[7] A disadvantage of xanthates is the risk they can pose to health and the environment. This risk is associated with the possibility of its decomposition, generating the CS_2 gas, which is highly toxic.[8]

It is important to note that a real aquatic environment has many metal species present, including alkaline, alkaline earth and heavy metals ions. Thus, when xanthate is used for capturing heavy metals ions, interference with other metal cations can occurs, namely with Na^+, Ca^{2+}, Mg^{2+}, K^+, among others. The nature of these metals influences the type of interaction with xanthate, which is a soft Pearson base. Thus, metals with a higher radius and/or small charge will interact better with xanthate due to the greater polarization of the electronic cloud. According to the acid/base hard/soft Pearson theory, it is possible to infer that metal ions with a higher charge concentration will interact electrostatically, differently of the soft acids that prefer to interact covalently with soft bases. In the case of heavy metals, a covalent interaction with xanthate will occur due to its soft acid character. In addition, as xanthate has two atoms capable of acting as a base, it is possible to infer that there will be a difference in basic character, in which the hard metal ions prefer to bind through the oxygen atom.[9]

2. Xanthates applications

Heavy metals are often found in industrial wastewater, electronic manufacturing, painting, fertilizers, battery manufacturing, and mining industry.[10] Metals, such as lead, cadmium and nickel are known for their nonbiodegradability and bioaccumulation, which make them very dangerous for humans, animals, and plants.[11] Exposure to heavy metals can cause several problems to human health, such as cancer, kidney problems, and death, in some cases.[12] The idea of using modified xanthates to remove heavy metals from aqueous environments is to generate a low-cost adsorbent, taking advantage of the properties of xanthates and cellulose present in the material (by-product), which would generally be discarded in the production processes. The use of these by-products makes modified xanthates a low-cost adsorbent.[13] In addition, there are several studies aimed at the use of xanthates for noblest purposes, with applications in the technological and biological areas. For example, Banti *et al.* reported the antitumor potential of silver(I) complexes with methyl xanthates for the human adenocarcinoma breast cancer cells.[14] In the technological area, Cheng Li *et al.*[15] studied the thermal decomposition of cadmium xanthate to produce CdS thin films.[15]

The environmental, biological, and technological applications reported in the literature in the last ten years are described in the following sections.

2.1. Environmental applications

Contamination of waters and wastewaters by heavy metals has been one of the biggest environmental problems due to the constant industrial development. Heavy metals

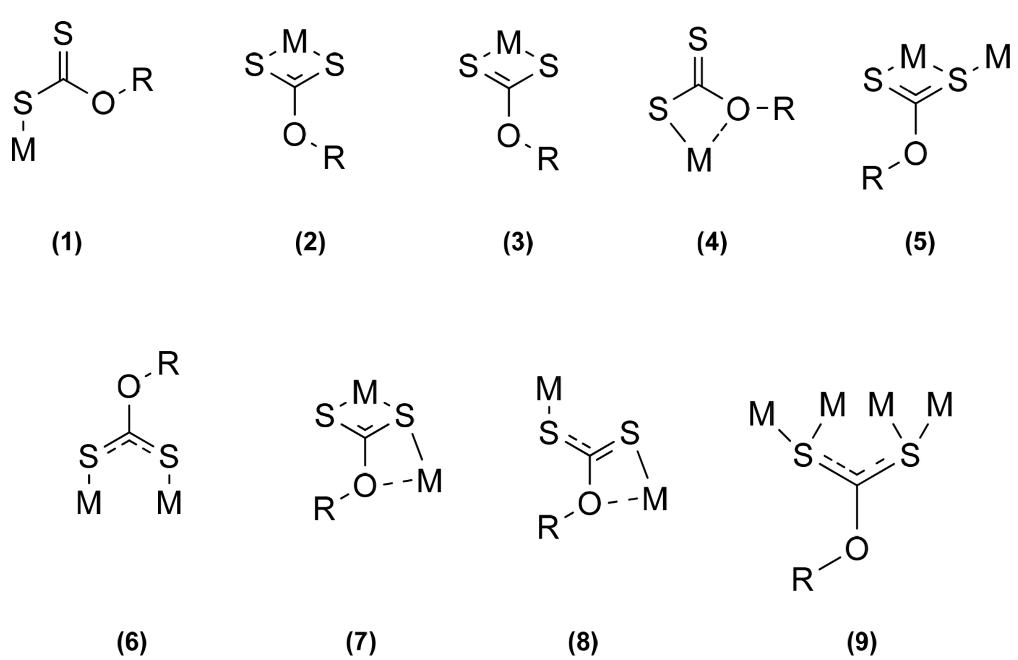


Figure 2. Different coordination modes of the xanthate ligands to metal centers[5]

ions of cadmium, lead, chromium, mercury, copper, and nickel are found in industrial effluents, such as in pigment manufacturing, galvanizing, electroplating, battery manufacturing, etc.[10,16]

These metals have been the subject of many environmental discussions, due to their toxicity, even in low concentrations, and the high demand from industries that dispose them in fresh water without previous treatment. Heavy metals are not metabolized by the body and accumulate in tissues, which increases the risk to human health. They can be introduced into the human body through water, food, and/or skin absorption.[7]

Nowadays, there are many ways to remove heavy metals from wastewater, such as flotation, adsorption, electrolytic, precipitation, and membrane processing methods, as highlighted in the review by Zeng *et al.*[17] In the adsorption technique, the use of by-products as adsorbents has attracted the attention of several researchers, mainly because of the low cost and high adsorption capacity.[14,18] Some articles report wastewater contamination by metals and the application of xanthate type ligands, including modified cellulose systems, to remove metal ions through adsorption, precipitation, and flotation techniques.[11-14]

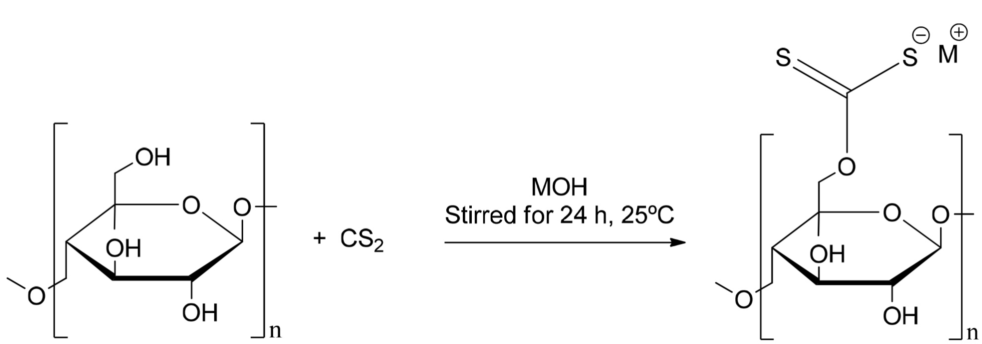
In the case of modified xanthates with cellulose, the final product, that will be used as an absorbent comes from the reaction of the cellulose present in the by-product with CS_2, as shown in Scheme 2.[19] In 2017, Wang *et al.*[11] modified a chitosan thiourea sponge with xanthate and combined it with *Pseudomonas putida I3* and *Talaromyces amestolkiae* to remove Pb^{2+} ions from aqueous solutions. This study proved the effectiveness in removing Pb(II) by xanthate chitosan. In the same year, Mohammed *et al.*[20] and Wang *et al.*[13] synthesized xanthates from starch sources. The xanthates synthesized by these research groups were used to adsorb Pb(II), Cd(II) and Cu(II) ions with satisfactory results.

Another interesting analysis can be made by comparing the removal of heavy metals in different water samples. An example of this kind of research was developed by Zhu *et al.*,[21] where the efficiency of a functionalized polyacrylamide (MPAM) xanthate system to remove Cr(VI) from different aqueous solutions, such as river, pool, and lake water, was evaluated. Another objective of the study was to test the possible interference of other cations, namely alkaline and alkaline earth ions. The removal efficiency of Cr(VI) was proportional to the dosage of MPAM with the best results observed when higher dosages of MPAM were used. Furthermore, it was verified that the presence of K^+, Ca^{2+} and Na^+ cations in the samples, inhibited the removal of Cr(VI) by xanthate.

The modified xanthate cellulose systems can also contribute to the reuse of disposable materials, such as by-products that often do not have an adequate discarding in nature. The works of Chand *et al.*[17], Qu *et al.*[18] and Nath *et al.*[22] address the synthesis of modified xanthates using apple pomace, rice husks and sugar, respectively. In these studies, the capacity of systems to remove heavy metals, such as Cd(II), Ni(II), Cu(II) and Pb(II) species from industrial wastewater systems, was evaluated. These cellulose systems showed good adsorption when compared to other adsorbents used in water treatment industries, which makes them promising and environmentally friendly.

In addition to cellulosic systems, xanthates have applications in the mining industries as flotation collectors. Flotation is a separation method widely used to separate valuable minerals. In flotation, the mineral surfaces can be changed using activators, collectors, modifiers or depressants agents.[23] Two studies conducted by Otero-Calvis *et al.*[23] and Zhang *et al.*[24] used alkyl xanthates as flotation collectors to separate heavy metals from synthetic aqueous solutions. In their study, Zhang *et al.*[24] highlighted the influence of pH on flotation through the flotation study of minerals galena and sphalerite, which are sources of zinc and lead, respectively. The pH is an important factor when using xanthates. At low pH, the predominant species are xanthic acid ($R\text{-}OCS_2H$). However, at a slightly higher pH, but still acidic, the decomposition of this species occurs, generating CS_2. At basic pH, the formation of CS_2 decreases until there is no more formation because this species will interact with the OH^- of the solution.[25]

Another important parameter is the performance of the collectors used in flotation. In order to explore the capacity of different xanthates, Yang *et al.*[26] studied the flotation of pyrite using amyl, isobutyl, and ethyl xanthates. The results showed that the capacity of the xanthates follows the order amyl xanthate > isobutyl xanthate > ethyl xanthate. This behavior can be explained by Pearson's acid-base theory, in which soft acids tend to interact with soft bases and hard acids interact with hard bases with higher formation



Scheme 2. Cellulose xanthate synthesis[12,19]

constants. In this pyrite-xanthate system, the alkyl xanthates act as Lewis bases, which are capable of donating electronic density. Among the alkyl xanthates studied, amyl xanthate has a better interaction with ferrous ion (a borderline Lewis acid) and ethyl xanthate interacts less. This effect is observed due to the size of the ligand chain in which larger carbon chains will donate more electron density to the oxygen atom, which will remove less electron density from the resonant site. Thus, xanthates with larger chains will have a higher electron density in the sulfur atoms to act as the Lewis base.[26] In addition, a flotation collector increases the hydrophilicity of the mineral's surface. In the case of xanthate use in the flotation process, oxidation can occur leading to a product that will be adsorbed on the mineral's surface. Therefore, xanthates with a larger chain make the surface of the mineral more hydrophilic than xanthates with smaller chains.[27] In this way, the order of capacity of the flotation collectors used can be explained.

2.2. Biological applications

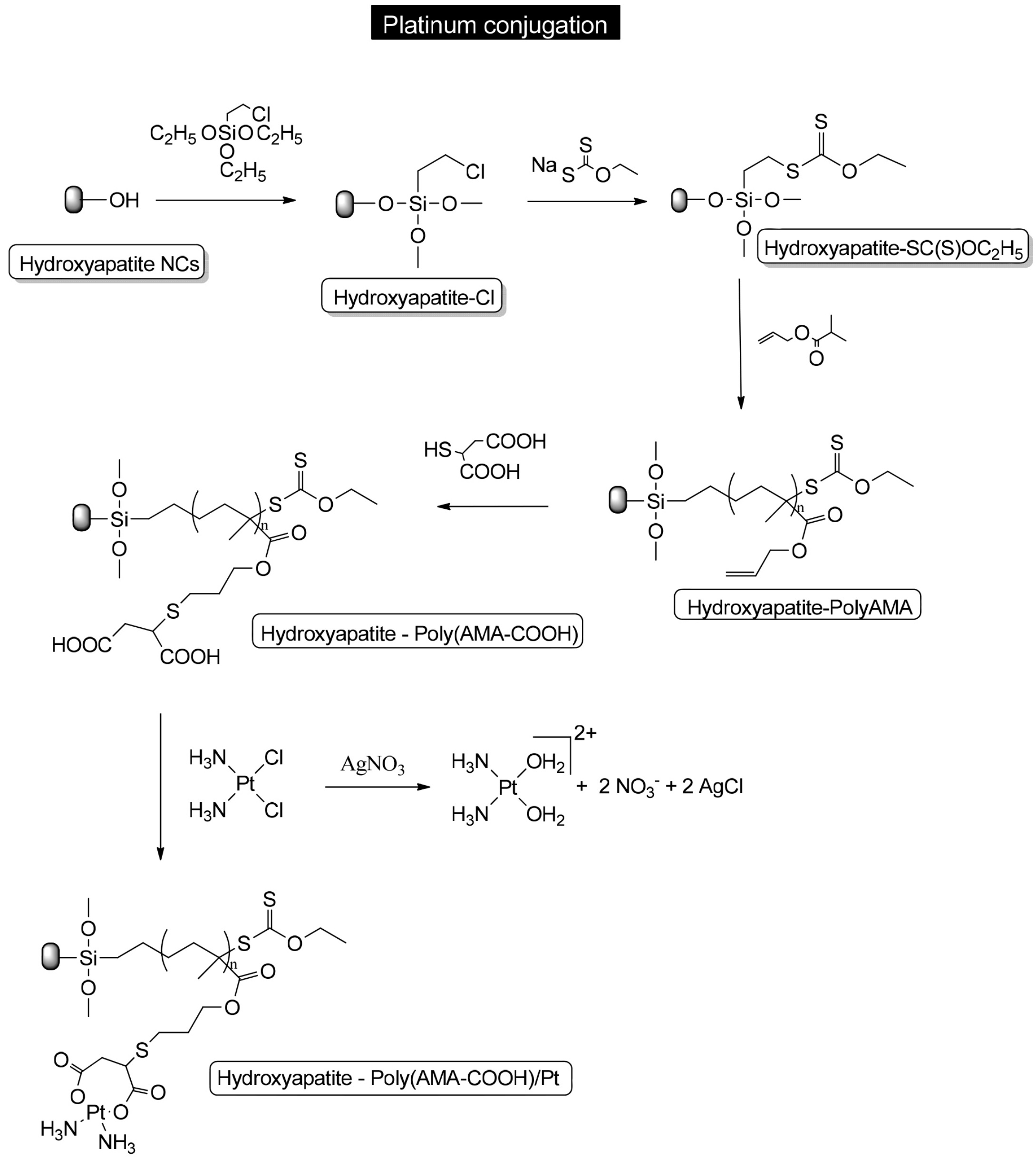
Xanthates show fungicide, pesticide and antibacterial activities.[28] The most used compound against bacteria is penicillin, which since its accidental discovery in 1928,[29] has been widely used as an antibiotic agent. However, with the continued use of penicillin-based drugs, many bacteria have acquired resistance to their derivatives. An example is *Staphylococcus aureus*, which is resistant to methicillin.[29] With the appearance of several bacteria resistant to the drugs already used, the search for new antibacterial agents is necessary. Several research groups[28,30-33] evaluated the antibacterial activities of the Ni(II), Fe(II), Sb(III) and V(III) complexes. The xanthate ligands used in the synthesis of the complexes were also evaluated in order to compare whether the complexation influences the antibacterial activity of these compounds. The research groups tested them against various bacteria, such as *Staphylococcus aureus*, *Escherichia coli*, *Pseudomas aeruginosa*, and *B. cereus*. The results of Heydari *et al.*[31] showed that all the ligands used (ethyl, propyl, butyl, pentyl, hexyl, heptyl and octyl xanthates) and the Fe(II) complexes were active against all the tested bacteria. The work of Qadir *et al.*,[28] Chauhan *et al.*[32] and Andotra *et al.*,[33] present heteroleptic complexes and only the $Na(4\text{-}Cl\text{-}3\text{-}CH_3C_6H_3OCS_2)$ used by the Andotra group showed activity against the bacterium *Staphylococcus aureus.* However, all complexes of Sb(III), Ni(II) and V(III) showed activity against all bacteria analyzed. When comparing the antibacterial activity of ligands and complexes, it is possible to conclude that the complexes were more active than the isolated ligands. This behavior can be explained by the chelation effect with the charge of the metal ion being reduced by neutralization with the sulfur charge present in the xanthate. In addition, chelation causes liposolubility to be greater in complexes, which facilitates the permeation of the bacteria cell membrane. The mechanism of action also includes the hydrophobic interaction of xanthates with cell constituents, interfering with the normal bacterial cell process.[31]

In addition, several studies involving xanthate complexes has shown antitumor activities. According to the World Health Organization (WHO), cancer is a set of diseases caused by abnormal cell growth.[34] Currently, chemotherapy is one of the most common treatments for cancer patients, among radiotherapy, surgery, and, more recently immunotherapy, hormonal and targeted therapy.[35,36] Among coordination compounds, the first important drug was cisplatin, which shows good results in several cancer lines (testicular, neck, bones, and ovarian cancers).[37] However, cisplatin causes several side effects in patients, which makes it necessary to search for other materials that may have comparable or superior anticancer activity. Since the discovery of this compound, several second-generation complexes have been tested for anticancer activity, entered in clinical trials and are worldwide or locally approved for treating cancer in humans.[35,38]

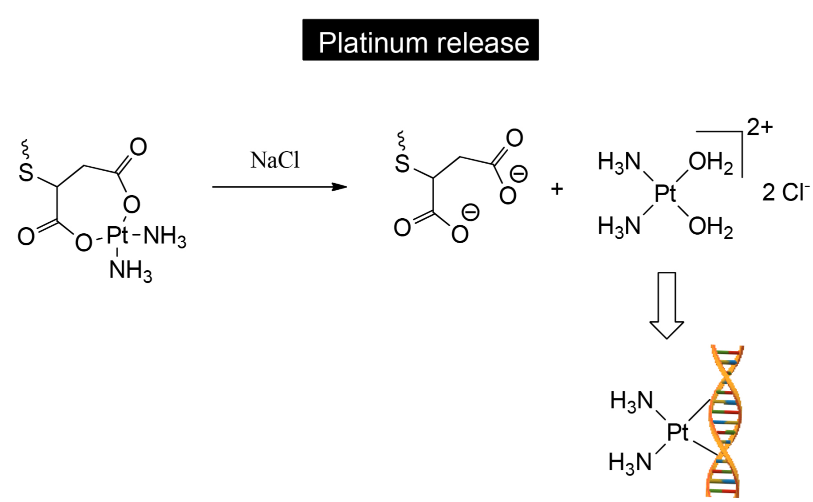
In addition to varying the metal ion, many studies have used several ligands that show good perspectives. There are studies in the literature that have shown good results for the use of complexes comprising ligands containing sulfur atoms, as is the case with xanthates. With the intention of reducing the toxic effects of cisplatin and increasing its antitumor activity, Bach *et al.*[39] developed a nanocarrier to transport the drug in the body. Ethyl xanthate was an important agent in the formation of the nanocarrier, as shown in Schemes 3 and 4.

The cytotoxic activities of the nanohybrid hydroxyapatite-poly(allylmethacrylate-COOH) (OHAp-poly(AMA-COOH)), the modified platinum complex and cisplatin were evaluated against epithelial cell lines (A549) and cervical cancer cells (HeLa) with 24, 48 and 72 h incubation times.[39] The results showed that OHAp-poly (AMA-COOH) is not active against both cell lines. In addition, it was observed that at longer incubation times, cisplatin and the modified complex were cytotoxic with the Pt(II) complex exhibiting lower cytotoxicity against HeLa cells than cisplatin after 24 h, but similar one after 48 h. This study predicts the reduction of side effects in the treatment of several cancers.[39]

Other studies have evaluated the cytotoxic activity of xanthate complexes[14,40-42] Sharma's group confirmed the cytotoxicity of Ni(II) complexes for breast (MCF-7), colon (HT-29, HCT-116 and SW-620), lung (A-549), ovary (OVCAR-5) and prostate (PC-3) cancer cells.[40] Among the synthesized complexes, $[Ni(m\text{-}C_2H_5C_6H_4OCS_2)_2(2\text{-}C_5H_4NCl)_2]$ showed excellent growth inhibition results for cell lines HT-29 (70%), HCT-116 (98%), SW- 620 (84%), A-549 (97%) and PC-3 (99%). These results indicate that the complex is promising mainly for the prostate cancer line (PC-3). Singh, Gandin and Banti groups synthesized xanthate complexes containing Co(II), Cu(II), Au(I) and Ag(I) metal centers.[14,41,42] The cytotoxicity of the complexes was measured by assessing the growth of inhibitory concentration (IC_{50}) against several cancer lines, such as



Scheme 3. Modification stages of cisplatin: Platinum conjugation[39]



Scheme 4. Modification stages of cisplatin: Platinum release[39]

skin (HEP3B), neuroblastoma (IMR-32), colon (HCT-15), ER positive breast (MCF-7) and ER negative (MDA-MB-231), lung (A549), and leukemia (HL60) (Table 1). The IC_{50} values show that some of the xanthate complexes exceed cisplatin activity, and are potential candidates in the treatment of cancer. The gold complex $[Au(EtOCS_2)(PEt_3)]$, synthesized by Gandin *et al.*, proved to be the more active in all cell lines tested.[42]

Another concern in the current cancer treatments is hypoxia in tumor tissue since the low oxygen concentration in these tissues decreases the tumor's response to radiotherapy. Currently, there are methods to detect tumor hypoxia, such as polarographic needle electrodes; however, this and other methods are invasive and cause more inconvenience to patients. Imaging techniques have been studied as a non-invasive alternative for detecting tumor hypoxia. In 2016, Li *et al.*[43] synthesized metronidazole xanthate (MNXT), which was radiolabeled with ^{99m}Tc-glucoheptonate (GX) and was used as a ligand for the synthesis of the ^{99m}TcO-MNXT complex. The ^{99m}TcO-MNXT complex was injected into female mice with mammary carcinoma (S180) and single photon emission computed tomography (SPECT) imaging studies were performed 4 h after the injection. A high accumulation of the complex in liver and kidneys has been reported, and further research is needed to verify possible side effects.

2.3. Technological applications

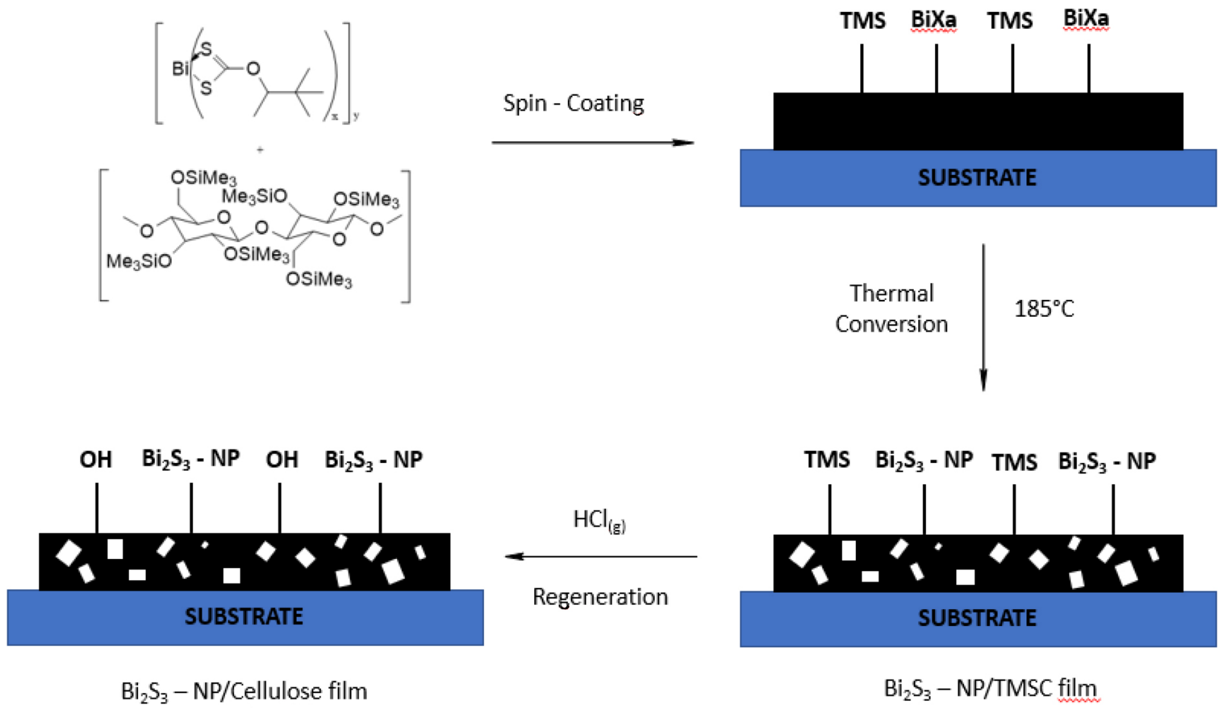
Humans have always created ways to use and improve materials from nature to facilitate and even prolong the life of their species. Technology consists of production in a rational way.[44] With the growth and development of cities, several needs arose, such as basic sanitation, creation of vaccines, and medicines, and even faster transportation facilities. New materials with technological applications have been developed over the years by researchers from different areas of science. From this perspective, some articles were selected that address the development of new technologies using xanthate complexes. Several research groups[45-49] conducted studies on thin films, involving Pb(II), Zn(II), Cu(I), Bi(III), and Co(II) xanthates. Thin films consist of nanomaterials that can be applied to substrates in order to modify their surface properties, such as optical, chemical and conductance.[48] There are several techniques for forming and depositing thin films, such as chemical vapor deposition, spray pyrolysis, deposition in chemical bath, spin-coating and thermal decomposition.[45] The most commonly used techniques for forming thin films have been thermal decomposition and spin-coating. The xanthate complexes are widely used in the manufacture of thin films due to their low cost, efficiency in the production of nanostructured metal sulfides and easy handling.[48] The thermal decomposition of xanthate complexes results in the formation of metal sulfides by pyrolysis reaction. This technique was applied by Wang *et al.*[45] and Onwudiwe *et al.*[46] to produce thin films of PbS, ZnS and, CdS. The PbS is a semiconductor that has been of great interest due to its diverse applications, such as infrared detectors, photoconductors, solar cells, etc. The CdS and ZnS nanoparticles synthesized aroused the interest of the Onwudiwe group[46] due to the optical properties that these materials present. The work developed by Reishofer *et al.*[48] clearly illustrates the formation of thin films on a substrate using the spin-coating technique (Scheme 5). The first stage of thin films formation, involves the Spin-coating technique on substrates for the formation of precursor films, starting from solutions of bismuth xanthate (BiXa) and cellulose (TMSC). The thermal conversion stage leads to the formation of thin nanocomposite bismuth / cellulose sulfide films after cleavage of the silyl groups with HCl.

The chemical bath deposition technique was performed in the work of Kariper *et al.*,[48] in which thin films of cobalt xanthate (CXTFs) were deposited on amorphous glass substrates. Thin films have been tested for their optical and electrical properties and have shown promise for use in detectors, sensors, and solar cells. Solar energy is a renewable energy source and is considered the most abundant and cleanest one. The most efficient way to benefit from solar energy is to convert it into electrical energy through solar cells. The application of thin films derived from xanthate complexes in solar cells was studied by several authors, including Fradler *et al.*,[50] Li *et al.*,[15] Wang *et al.*,[51] and Nam *et al.*[52]

Table 1. IC_{50} values of xanthate complexes for different cell lines[15,41,42]

Compound	IC_{50} (µM) ± S.D.							References
	HEP3B	IMR-32	HCT-15	MCF-7	MDA-MB-231	A549	HL60	
$[Au(EtOCS_2)(PEt_3)]$[(a)]	-	-	0.61±0.13	0.41±0.21	-	0.33±0.47	0.19±0.06	42
$[K_2(COS_2\text{-bz-Inamin})]$[(b)]	17.8±2.9	23.2±1.4	-	-	-	-	-	41
$[K_2(COS_2)(\text{naphmetInamin})]$[(c)]	79.5±6.1	26.2±1.3	-	-	-	-	-	41
$[Ag(CH_3OCS_2)(tpp)_2]$[(d)]	-	-	-	> 30	11.6±1.0	-	-	14
$[Ag(CH_3OCS_2)(tptp)_2]$[(e)]	-	-	-	20.0±1.3	> 30	-	-	14
$[Ag(CH_3OCS_2)(tmtp)_2]$[(f)]	-	-	-	12.3±0.3	> 30	-	-	14
Cisplatin	74.6±1.3	107±0.3	20.34±1.31	19.04±1.5	26.7±1.1	29.21±1.92	4.57±1.13	14, 41, 42

[(a)]O-Ethyl carbonodithioato-κS)(triethylphosphine)gold(I); [(b)]K_2[4,4'-bis(*2*-dithiocarbonatobenzylideneamino)diphenyl ether]; [(c)]K_2[4,4'-bis(*2*-dithiocarbonatonaphthylmethylideneamino)diphenyl ether]; [(d)]tpp = triphenylphoshine [(e)]tptp = tri(p-tolyl)phosphine; [(f)]tmtp = tri(m-tolyl)phosphine



Scheme 5. Formation of thin films by the Spin-Coating technique[48]

Another technological application of the xanthate complexes has been their use as catalysts in the curing process of epoxy resins. Such process consists of the transformation of the low molecular weight polymer or epoxy monomer into a three-dimensional network. The catalysts can be basic, such as amines and anhydrides or acids, such as BF_3 or $AlCl_3$. The main disadvantage of conventional catalysts is the shortening of the pot-life. An alternative that does not present this disadvantage is the use of latent healing catalysts, which act only when external stimuli are provided. In the work developed by Vagvala *et al.*,[53] xanthate complexes of Cu(I), Pb(II), Sb(III), Zn(II), Cd(II), In(III), and Ga(III) containing branched and linear chains were synthesized. Subsequently, an investigation of epoxy resin curing was carried out and the results showed that the curing time of the epoxy resin increased with the increase of the alkyl chain size for the linear xanthate complexes. For those with branched chains, it was observed that increasing the chain decreased the healing time. In addition, the curing time was evaluated at different temperatures of thermal decomposition, showing that the lowest temperature had the shortest curing time. Among the complexes evaluated, Ga(III) xanthate was the most efficient because it had the lowest decomposition temperature. Due to the possibility of adjusting these parameters, the xanthate complexes are promising for the control of the epoxy cure.[53]

3. Final Considerations

This review was based on studies that addressed applications of xanthates and their complexes in the last ten years. Although the xanthate class was discovered in the 19th century, this document shows that its applications are still important and widely explored nowadays, since its low production costs and its simple preparation methods. The various studies explored in the environmental, biological, and technological areas confirmed the observed potentialities. The well-known flocculation property broadly discussed in the articles addressed has gained new expansion with the exploration of cellulosic materials for the removal of heavy metals from wastewater. In their biological applications, xanthate complexes have been shown to be effective against different cancer cell lines and various bacteria, thus being one more tool in combating different diseases. Finally, xanthate complexes have been shown to be relevant for technological applications, such as the production of thin films and their application in the manufacture of solar cells as well as in the control of the epoxy cure.

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