

**STUDY ON WATER-SOLUBLE POLYMERS FOR
BIOMEDICAL APPLICATIONS**

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**STUDY ON WATER-SOLUBLE POLYMERS FOR BIOMEDICAL
APPLICATIONS**

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DECLARATION

I hereby declare that I have conducted, completed the research work and written the dissertation entitled 'Study on Water-Soluble Polymers for Biomedical Applications'. I also declare that it has not been previously submitted for the award of any degree and diploma or other similar title of this for any other examining body or University.

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LIST OF SYMBOLS

A	Absorbance
c	molar concentration
l	optical path length
m_0	Initial mass of the sample at hour 0
m_t	Mass at the extraction hour
Mw	Molecular weight
T_m	Melting point
X_c	Crystallinity
ΔH	Measured melting enthalpy of the polymer sample
$\Delta H_{100\%}$	Theoretical melting enthalpy of 100% crystalline PVOH
ε	molar absorption coefficient
Φ	Residual weight fraction

LIST OF ABBREVIATIONS

AA	Acetic acid
AAS	Atomic absorption spectroscopy
ASTM	The American Society for Testing and Materials
ATP	Adenosine triphosphate
BKC	Benzalkonium chloride
CF	Cellulose fiber
CMC	Carboxymethylcellulose
CNC	Cellulose nanocrystal
CNF	Cellulose nanofibril
DMSO	Dimethyl sulfoxide
DSC	Differential scanning calorimetry
EDTA	Ethylenediaminetetraacetic acid
FTIR	Fourier transform infrared spectroscopy
GPPS	General-purpose polystyrene
HAI	Hospital-acquired infection
HEC	Hydroxyethyl cellulose
ISO	International Organization for Standardization
LLDPE	Linear low density polyethylene
MWCNT	Multi-walled carbon nanotube
NCC	Nanocrystalline cellulose
nd	Not determined
PA	Polyamide
PAA	Polyacrylamide
PE	Polyethylene
PEG	Polyethylene glycol
PET	Polyethylene terephthalate
PICC	Peripherally inserted central catheter
PLLA	Poly(L-lactide)
PP	Polypropylene
PS	Polystyrene
PVA	Polyvinyl alcohol

PVC	Polyvinyl chloride
PVOH	Polyvinyl alcohol
RLU	Relative light unit
SEM	Scanning electron microscope
TPU	Thermoplastic polyurethane
USM	Universiti Sains Malaysia
UTM	Universal testing machine
UTS	Ultimate tensile strength
UV-Vis	Ultraviolet-visible

LIST OF APPENDICES

Appendix A

STUDY ON WATER-SOLUBLE POLYMERS FOR BIOMEDICAL APPLICATIONS

ABSTRAK

Produk polimer mempunyai banyak sifat menarik yang menjadikan mereka diguna secara meluas dalam kehidupan seharian. Namun, keburukan polimer ialah pengumpulan sisa plastic secara global. Dalam kajian ini, polimer larut air dikaji untuk menghasilkan tiub plastic yang berongga bagi kegunaan pengumpul PI yang berfungsi untuk mengumpul air kencing dalam bidang kesihatan. Sifat struktur, kimia, mekanikal, terma dan kadar kelarutan tiub plastik dikaji. Selain itu, tong pengumpul PI tertakluk kepada pertumbuhan mikrob, bau air kencing dan mendakan kalsium yang akan merosakkan pengumpul PI. Oleh itu, filem larut air yang menyekat pertumbuhan mikrob, mempunyai deodoran untuk mengurangkan bau dan penyahkalsifikasi untuk memerangkap kalsium dihasilkan. Beberapa teknik pencirian termasuk FTIR, ujian tegangan, DSC, ujian kebenjolan dan kelarutan bagi fasa satu manakala AAS, ujian hidu dan ujian ATP bagi fasa dua dijalankan. Dalam fasa satu, suhu penyemperitan optimum bagi tiub PVOH di zon muatan/zon leburan/zon acuan ialah 185/201/203/182°C dengan kelajuan skru sebanyak 7 rpm. Kekuatan tegangan tiub ialah 11.38 MPa dan pemanjangan sebanyak 1884.33% telah dicapai. Tiub PVOH merekodkan titik cair pada 283.19°C. Sepanjang rendaman dalam air, tiub membengkak di suhu bilik dan 40°C sebelum melarut selepas 6 dan 4 jam masing-masing. Manakala, tiub melarut sepenuhnya selepas 3 dan 2 jam rendaman dalam air yang bersuhu 50°C dan 60°C. Tiub PVOH mencatatkan kadar larutan yang paling tinggi sebanyak 0.03903 mol/L h pada 60°C. Dalam fasa dua, 4 ml AA digunakan untuk menyahkalsifikasikan air kencing tiruan manakala 5 ml BKC berkesan untuk mengurangkan kiraan bahan cemar dan bau air kencing tiruan.

STUDY ON WATER-SOLUBLE POLYMERS FOR BIOMEDICAL APPLICATIONS

ABSTRACT

Polymeric products have many remarkable properties which make them to be widely used in daily life. However, the downside of polymers is the accumulation of plastic waste globally. In this research, water-soluble polymers were investigated for a hollow plastic tube for the PI collector; which is used to collect urine in healthcare segment. The structural, chemical, mechanical, thermal properties and rate of solubility of the tubes are investigated. Apart from that, the PI collector tank is also subjected to microbes' growth, urine odor and calcium precipitation which may potentially damage the PI collector. Thus, we intend to fabricate a soluble polymer film that is antiseptic in nature to suppress the microbes' growth, contains a deodorant to reduce the smell and has a decalcifier to chelate and trap the calcium salt. Several characterization techniques, including FTIR, tensile testing, DSC, swelling and dissolution studies for phase one whereas AAS, physical smelling and ATP test for phase two were performed. In phase one, the optimized extrusion temperatures at the feed zone/melting zone/die zone of the extruder for PVOH tubes were 185/201/203/182°C respectively with a screw speed of 7 rpm. The average tensile stress and elongation at break of the polymeric tube is 11.38 MPa and 1884.33% respectively. The PVOH tube recorded a melting point of 283.19°C. When immersing in tap water, the tubes swelled up at room temperature and 40°C before dissolving after 6 and 4 hours respectively. Whereas, the tubes dissolved completely after 3 and 2 hours of immersion at 50°C and 60°C. At 60°C, the PVOH tube showed the highest dissolution rate of 0.03903 mol/L h. In phase two, 4 ml of AA to decalcify the

artificial urine whereas 5 ml of BKC is effective to reduce the contaminants count as well as deodorize the artificial urine.

CHAPTER 1

INTRODUCTION

1.1 Research Background

There are two types of polymers, namely natural and synthetic polymers. Cellulose and proteins are examples of natural ones. On the other hand, the first synthesized polymer was Bakelite being produced in 1907 (Eriksen *et al.*, 2014). It marked the breakthrough to manufacture other polymers such as polyamide (PA), polyethylene (PE), polyethylene terephthalate (PET), polypropylene (PP), polystyrene (PS) and polyvinyl chloride (PVC). Polymeric materials exhibit significant properties such as excellent barrier and mechanical properties, easy processability, relatively low cost and large availability of synthetic polymers (Abdullah *et al.*, 2017), resulting in extensive use in daily applications. For example, polycarbonate for eyeglasses and compact discs, polyesters for fabrics and textiles, polyvinylidene chloride for food packaging, etc. (Tiseo, 2022).

Referring to Figure 1.1, since 1950s, the global production of plastic has experienced rapid growth. There were around 9.2 billion tons of plastics produced since 1950. It was estimated that the global plastic production reached 367 million metric tons in 2020 (Tiseo, 2022). According to Dent (2020), it is forecasted that the global plastic production is going to reach 600 million tons by 2030.

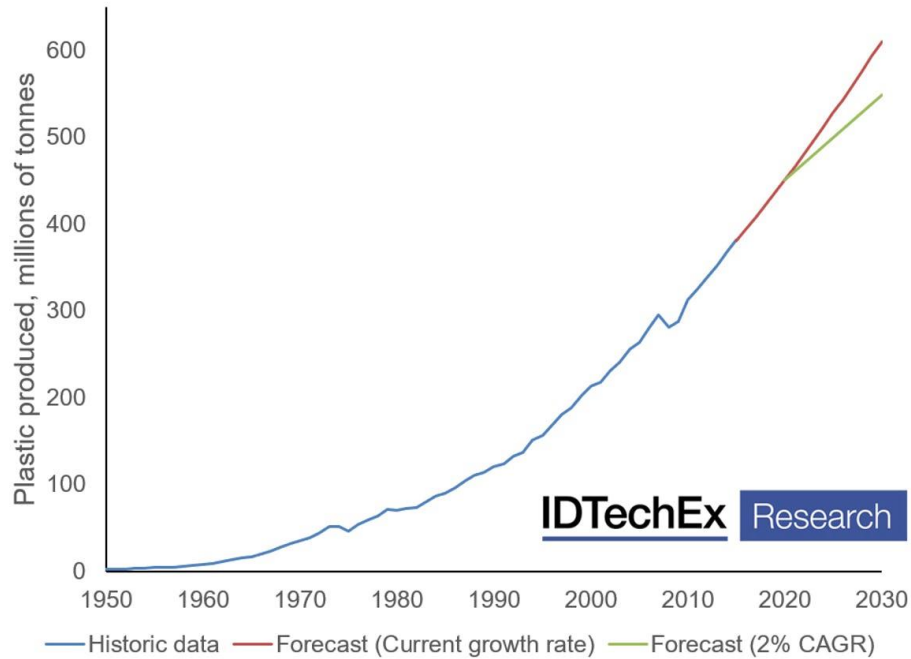


Figure 1.1 Global plastic production from year 1950 to 2030 (Dent, 2020)

According to UNEP (2021), to date, it is reported that only approximately 30% of these plastics remain in use while the remaining portion was generated as the primary plastic waste around the globe. It is estimated that less than 10% of the global plastic waste produced between 1950 and 2017 have undergone recycling, while the remaining 14% has been incinerated and 76% has been discarded in landfills or discharged into the environment, including the oceans (UNEP, 2021). Due to the steady increase in the manufacture of plastic products, driven by consumer demand, together with the inadequate waste management improvements, it is estimated that each year an amount of 23 to 37 million tons of plastic waste could end up at the oceans by 2040 and 155 to 265 million tons could be released into the environment by 2060.

To solve the plastic pollution issue, water-soluble polymers are introduced into the world. The popular water-soluble polymers found in the global market include polyacrylamide, polyvinyl alcohol, guar gum, cellulose ether, gelatin, xanthan gum, casein, polyacrylic acid, etc. Water-soluble polymers are being used in a wide variety of applications such as food and beverages, detergents and personal care, oil and gas,

pharmaceutical, pulp and paper as well as water treatment sector. The widespread use of water-soluble polymers is due to their unique properties, including environment-friendly nature, fast drying, inflammable and mild odor which make water-soluble polymers grow fast globally. By the year 2022, it is expected that the value of the global water-soluble polymer market to be at USD 43.79 billion (Manuel, 2016).

One of the important market segments that requires water-soluble polymers is healthcare. This is because hospital plastic waste is classified as biohazard waste and must be incinerated at a cost of RM5.20 per kg. As a result, the healthcare segment is proactively investigating the possibility of using a water-soluble plastic tube instead of a polyolefin hollow plastic tube for medical appliances. One of the projects offered to Universiti Sains Malaysia (USM) is a water-soluble tube for the PI collector (Figure 1.2). This tube is required for proper urine collection using PI collector to avoid current manual practices which cause the spread of hospital-acquired infections (HAIs). At present, a nurse manually drains urine from the urine bag into a pail and walks to the toilet to dispose of the waste. This method is indiscreet, unhygienic and dangerous for it raises the risk of contracting infections both to the nurse and patients.



Figure 1.2 A PI collector

The PI collector is a portable device that can collect urine discreetly, safely and hygienically from ten consecutive bags and print the volume of urine drained. After a single collection trip, the urine is effectively disposed of. The nurse is only required to hook on then remove the device once it has been entirely drained. The purpose is to minimize HAIs that burden insurance companies and patients while increasing work efficiency and lowering hospital disposal costs.

Therefore, the aim of this project is basically to fabricate a water-soluble hollow plastic tube for the PI collector. The rate of solubility and structural, chemical, thermal and mechanical properties of the tubes are investigated. Apart from that, the PI collector tank is also subjected to microbes' growth, urine odor and calcium precipitation which will coat the walls, tubes and motor that may potentially damage the PI collector. Thus, we intend to fabricate a soluble polymer film that is antiseptic in nature to suppress the microbes' growth, contains a deodorant to reduce the smell and has a decalcifier to chelate and trap the calcium salts.

1.2 Problem Statement

1.2.1 Extrusion Parameters for Water-Soluble Polymers

Polyacrylamide, polyacrylic acid copolymer, polyethylene glycol (PEG), and polyvinyl alcohol (PVA) are several examples of water-soluble polymers being found in the global market (Moja, Mishra and Mishra, 2021). For instance, PEG 6000 shows a complete solubility of 50 mg/ml in water at 20°C (Sigma-Aldrich, 2018). Furthermore, PVA that has a 88% degree of hydrolysis and a degree of polymerization of 1600 to 2000 also exhibits 100% water solubility (Mali *et al.*, 2019). Some of these water-soluble polymers like polyacrylamides are possible to be extruded. However, the extrusion of such water-soluble polymers in a tubular form is not well reported. There are difficulties

present during the extrusion process, for example, water-soluble polymers will dissolve when they are in contact with the water-cooling tank of the extruder (Herrild, 2020). Furthermore, the temperature as well as the screw speed for each type of polymer at which extrusion is performed is not well established. The barrel temperature during the extrusion process is to be determined via trial-and-error experiments (Resonnek and Schöppner, 2019). Thus, polyvinyl alcohol (PVOH) is introduced in this project for it is capable to be extruded at a higher temperature, despite its good solubility in water (Kuraray Poval, 2020). Nevertheless, due to lack of established extrusion parameters for PVOH, the temperature at the feed zone, melting zone and die zone as well as the screw speed of the extruder are also studied in order to fabricate a good hollow plastic tube.

1.2.2 Issues with Human Urine Collected in PI Collector

As stated in Section 1.1, a PI collector tank which is used to collect urine before disposal is potentially be damaged by the urine contained in the tank as the tank is exposed to the growth of microbes, unpleasant smell of urine and precipitation of calcium that gradually coat the walls, tubes and motor of the PI collector. To date, the urinary microbiota consisting of more than 100 species from exceeding 50 genera are inhabiting the human urinary tract as well as urogenital tract (Neugent *et al.*, 2020). It is reported that human urine comprises of an average of 5.8 bacterial species in females whereas 7.1 bacterial species were found in males (Nall, 2021). Plus, urine has an unpleasant smell when it leaves the human body. Although 95% of the urine is made up of water, it consists of urea, chloride, sulphate and other ions and molecules. Human that age from 18 to 77 years old contain 250 mg and 200 mg of calcium ion per day in their urine for males and females respectively (Sarigul, Korkmaz and Kurultak, 2019). After the urination process by utilizing the PI collector, the presence of bacteria, urine odor and calcium in the tank will cause HAIs in the wards as well as damaging the collector. Therefore, to sustain the

PI collector shelf life, it is essential to reduce microbes count, odor and calcium content. Although there are papers reporting on the use of titanium dioxide (TiO_2) and hydrogen peroxide (H_2O_2) as deodorants, ethylenediaminetetraacetic acid (EDTA) and acetic acid (AA) as decalcifiers and glutaraldehyde and benzalkonium chloride (BKC) as antiseptic chemicals, the use of antiseptic, decalcification and deodorization chemicals in one combined form using water-soluble polymers as the matrix is not well reported. Plus, the combination of chemicals in PVA films which aims to ease the applications in the hospital environment is not reported too. Hence, in this study, a 2-in-1 sachet containing a combination of antiseptic, deodorization and decalcification properties is fabricated in the form of films and the functionality is characterized to reduce the number of bacteria, unpleasant odor and level of calcium in the urine.

1.3 Objectives

There are three objectives in this research:

- i. To investigate the possibility of fabricating water-soluble polymer tubes using two different water-soluble polymers (PVA and PVOH) by extrusion method for PI collector system.
- ii. To evaluate the structural, chemical, mechanical, thermal properties and dissolution rate of the well-formed tubes.
- iii. To investigate the water-soluble polymer films that possess antiseptic, deodorization, and decalcification properties fabricated by solution casting method.

1.4 Scope of Study

In this research, PVA and PVOH were selected to fabricate into a tube structure via extrusion method. During the extrusion process, the temperatures at the feed zone,

melting zone and die zone as well as the screw speed of the extruder were varied accordingly. The extrusion temperatures and screw speed were stretched from 180 to 205°C and ranged from 5 to 7 rpm respectively before a good PVA or PVOH hollow tube could be extruded. After the well-structured tube has formed, the tube was characterized to understand the structural, chemical, mechanical and thermal properties. Fourier transform infrared spectroscopy (FTIR) was used to identify the functional groups in the tubes and the mechanical properties were characterized using a universal tensile machine to determine their tensile strength and elongation at break. The thermal analysis of the polymeric tube was also performed using differential scanning calorimetry (DSC) to identify its melting point. Furthermore, swelling studies were carried out for the polymeric tubes by measuring their weight in terms of weight increment. In addition, the dissolution rate of the tube in an artificial urine medium was also investigated to determine the lifetime of the tube during the urine transfer process. For this, the polymeric tube was soaked in artificial urine and the weight loss of the tube was determined. For functional studies, the tubes were immersed in tap water with different temperatures to study the dissolution rate of the polymeric tubes. The dissolution rate was evaluated using ultraviolet-visible (UV-Vis) spectroscopy.

In addition, a 2-in-1 sachet in the form of a film which possesses antiseptic, deodorization and decalcification properties was fabricated by solution casting method. The films containing different amount of acetic acid (AA) that ranged from 4 to 16 ml and benzalkonium chloride (BKC) which was varied from 1 to 5 ml were studied to determine the optimum amount of chemicals that was essential in functioning as the 2-in-1 sachet. PVA functioned as the matrix to enhance the dissolution of the sachets in the urine medium was comprised of AA and BKC. The antiseptic, deodorization and decalcification properties were quantified by using an Adenosine triphosphate (ATP)

tester, physical smelling and reduction of calcium through atomic absorption spectroscopy (AAS) respectively.

1.5 Thesis Outline

There are 5 chapters comprised in this thesis. Chapter 1 denotes the present circumstances and topic of plastic products and the development of water-soluble polymers. The drawbacks regarding the rising water-soluble polymers were highlighted. In this research, water-soluble hollow plastics tubes that were to be connected to a PI collector for the purpose of urine collection and disposal in the hospital ward in order to reduce the risk of hospital-acquired infections (HAIs) were presented. Furthermore, soluble polymer films to be placed into the PI collector tank that can exhibit antiseptic, deodorization and decalcification properties were highlighted too. In addition, Chapter 1 also presents the objectives of this research, scope of study and thesis outline. Chapter 2 depicts the literature reviews on the research associated with water soluble polymers carried out in the past. The definition of water-soluble polymers as well as its classification were presented. The dissolution mechanism, processing of polymers and parameters related to solubility were discussed too. Besides, the mechanical properties were also studied. Plus, this chapter also includes a topic on human urine. The chemicals that show deodorization, decalcification and antiseptic properties were presented. Chapter 3 shows the materials, fabrication and characterization methods used in this research to produce water soluble polymer tubes from PVOH as well as films by using PVA. Chapter 4 discusses the results obtained from the experiments carried out in this project. Chapter 5 summarizes the highlights of the study and the general conclusion of the research. Suggestions and recommendations for future studies were also incorporated in this chapter.

CHAPTER 2

LITERATURE REVIEW

2.1 Water-Soluble Polymers

Water-soluble polymers such as polyacrylamides, polyacrylic acid copolymer, polyethylene glycol and polyvinyl alcohol are materials that dissolve, disperse or swell in water (Kadajji and Betageri, 2011; Moja, Mishra and Mishra, 2021). Typically, the polymers will have repeating units or blocks of units in which there are hydrophilic groups comprised in the backbone of the polymer chains (Kadajji and Betageri, 2011). The properties of water-soluble polymers include quick drying, non-flammable, possessing mild odor and being more environmentally acceptable than any other polymers (Niir Project Consultancy Services Delhi, 2017).

Water-soluble polymers have an extensive function in aqueous media, including binders, dispersing and suspending agents, film-formers, flocculants and coagulants, humectants, lubricants, stabilizers, and thickeners (Moja, Mishra and Mishra, 2021). Furthermore, water-soluble polymers have a wide variety of industrial applications such as adhesives, coatings, constructions, food, paint, paper, pharmaceuticals, textile, water treatment, etc. (Kadajji and Betageri, 2011).

Water-soluble polymers can be divided into three categories, namely natural, semisynthetic and synthetic polymers. Natural water-soluble polymers comprise of microbial-, plant- and animal-derived materials. Semisynthetic polymers are those being manufactured by chemical derivation of natural organic materials whereas synthetic polymers are produced by the polymerization of monomers synthesized from petroleum- or natural gas-derived raw materials. Table 2.1 summarizes the advantages and disadvantages of various categories of water-soluble polymers as well as their water

solubility and extrusion characteristics and their details are reviewed in the subsequent section.

Table 2.1 Summary of the advantages and disadvantages of various categories of water-soluble polymers

Categories	Examples	Advantages	Disadvantages	Solubility in water	Extrusion criteria	References
Natural	Pectin	<ul style="list-style-type: none"> • Non-toxic 	<ul style="list-style-type: none"> • Require a lot of enzymes to synthesize due to structure complexity • Low tensile strength and elongation at break values 	Soluble in hot water but slowly soluble in cold water	Extrudable with the addition of starch	(Mukhiddinov <i>et al.</i> , 2000; Kumar, Mishra and Banthia, 2010; Kadajji and Betageri, 2011; Yadav <i>et al.</i> , 2017; Lara-Espinoza <i>et al.</i> , 2018; Elizabeth, Marcelo and Alejandra, 2019; Mada, Duraisamy and Guesh, 2022)
	Xanthan gum	<ul style="list-style-type: none"> • Stable to pH changes • Enhanced tensile strength of chitosan films 	<ul style="list-style-type: none"> • High molecular weight • High production cost 	Soluble in both hot and cold water	Extrudable and disperse in water with no lumps	(Carignatto <i>et al.</i> , 2011; Kadajji and Betageri, 2011; de Morais Lima <i>et al.</i> , 2017; Jindal and Singh Khattar, 2018; Pathak <i>et al.</i> , 2021; Zeng <i>et al.</i> , 2021)
Semisynthetic	Carboxymethylcellulose (CMC)	<ul style="list-style-type: none"> • Good film forming properties • Non-toxic 	<ul style="list-style-type: none"> • Poor mechanical properties 	Soluble in water at any temperature	Show a lubricating action towards the	(Boruvkova and Wiener, 2011; Aravamudhan <i>et al.</i> , 2014; Rani <i>et</i>

		<ul style="list-style-type: none"> • Biocompatible • Biodegradable 	<ul style="list-style-type: none"> • Low resistance to insects, light and rot 		extrusion process	<i>al.</i> , 2014; Ergun, Guo and Huebner-Keese, 2016; Veronese <i>et al.</i> , 2018; Noreen <i>et al.</i> , 2020; Kontogiorgos, 2022)
	Hydroxyethyl cellulose (HEC)	<ul style="list-style-type: none"> • Non-toxic 	<ul style="list-style-type: none"> • Agglomerate when in contact with water • Subjected to biological attack • Relatively weak mechanical properties 	Soluble in both hot and cold water	Extrudable by adding cellulose fibers	(Polymerdatabase, no date; Di Giuseppe, 2018; Taheri, Hietala and Oksman, 2020; Fortune Business Insights, 2021; Huang <i>et al.</i> , 2021)
Synthetic	Polyacrylamide (PAA)	<ul style="list-style-type: none"> • Non-toxic • Stable to pH changes 	<ul style="list-style-type: none"> • Easy degraded by mechanical action and oxygen • Made up of monomers (acrylamide) that are carcinogenic and neurotoxic 	Soluble in water	Extrudable starch-PAA graft copolymer	(Doble and Kumar, 2005; Willett and Finkenstadt, 2006; Kadajji and Betageri, 2011; Herth, Schornick and Buchholz, 2015; ChemicalBook, 2016; Fortune Business Insights, 2021)

	Polyvinyl alcohol (PVA)	<ul style="list-style-type: none"> • Non-toxic • Biocompatible • Biodegradable • Good film forming properties • Chemical stability • Flexibility • High hydrophilic properties • Gas barrier properties 	<ul style="list-style-type: none"> • Poor mechanical properties • Release toxic vapors if overheated • Expensive 	100% water solubility (PVA with 88% degree of hydrolysis and a degree of polymerization of 1600 to 2000)	Non-extrudable due to formation of hydrogen bonds inside its crystals	(Zhu <i>et al.</i> , 2013; Samzadeh-Kermani and Esfandiary, 2016; Gobbato, 2019; Harpaz <i>et al.</i> , 2019; Kassab <i>et al.</i> , 2019; Mali <i>et al.</i> , 2019; Dhall and Alam, 2020; Singh <i>et al.</i> , 2021)
	Polyvinyl alcohol (PVOH)	<ul style="list-style-type: none"> • Presence of acetate groups that enhance water solubility • Biodegradable • Non-carcinogenic • Non-toxic • High tensile strength 	<ul style="list-style-type: none"> • Presence of hydroxyl groups that induce inter and intramolecular hydrogen bonding that slow down water solubility 	100% water solubility at 80°C (PVOH with 97% degree of hydrolysis)	Extrudable at a high temperature due to crystalline nature	(De Jaeghere <i>et al.</i> , 2015; Kuraray Poval, 2020)

2.1.1 Pectin

Pectin is comprised of polysaccharides and mainly found in fruits such as apple pomades or citrus peel (Kadajji and Betageri, 2011). It contains D-galacturonic acid (GalA) units (Mukhiddinov *et al.*, 2000) that are chained by α -(1-4) glycosidic linkage. There are carboxyl groups found in the uronic acids in which some of the carboxyl groups are naturally present as methyl esters whereas some are commercially treated with ammonia to produce carboxamide groups (Figure 2.1). Pectin is non-toxic (Yadav *et al.*, 2017) and it has complete solubility in hot water but it is slowly soluble in cold water (Mada, Duraisamy and Guesh, 2022). However, to synthesize pectin, many enzymes are required due to the complexity of pectin structures (Lara-Espinoza *et al.*, 2018). Plus, pectin shows a low value of tensile strength and elongation at break (Kumar, Mishra and Banthia, 2010). It is concluded that the extrusion of pectin was successfully fulfilled by adding starch as the presence of starch made the extrusion process to be operated at a higher temperature compared to the processing of pectin alone (Elizabeth, Marcelo and Alejandra, 2019). Nevertheless, it has been widely used in the pharmaceutical industry. For instance, pure and standardized pectin is used as a binding agent in tablets as well as being a matrix for the entrapment and delivery of various drugs, proteins and cells (Kadajji and Betageri, 2011).

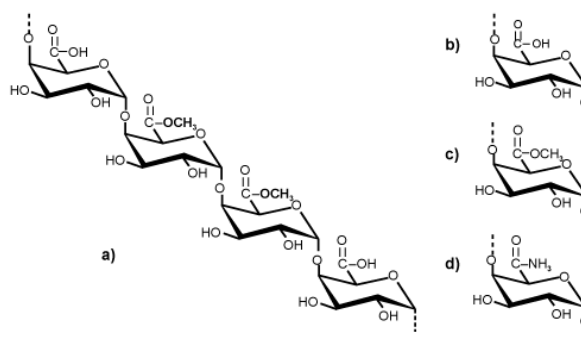


Figure 2.1 (a) A repeating segment of pectin molecule and functional groups; (b) carboxyl; (c) ester; (d) amide in pectin chain (Sriamornsak, 2003)

2.1.2 Xanthan Gum

Xanthan gum is a high molecular weight heteropolysaccharide secreted by the microorganisms *Xanthomonas campestris* (Pathak et al., 2021). However, the internationally growth medium for *X. campestris* which is the yeast malt medium is expensive (Carignatto et al., 2011), resulting in the high production cost of xanthan gum. Primarily, xanthan is made up of repeating pentasaccharide units comprising of two D-glucopyranosyl units, two D-mannopyranosyl units and one D-glucopyranosyluronic unit. Xanthan is soluble in both hot and cold water. It also has good stability in which xanthan are stable in both acidic and alkaline conditions and least influenced by the changes in pH. It is being used as a stabilizer and thickener as it offers high viscous solutions at low concentrations (Kadajji and Betageri, 2011). It is reported that the addition of xanthan gum into the films made with chitosan in the proportion of 50:50 enhanced the tensile strength of the films by approximately 2-fold (de Moraes Lima et al., 2017). In addition, Zeng et al. (2021) reported that xanthan gum could be extruded. It dispersed in water with no lumps being produced (Zeng et al., 2021). It is widely applied in personal care products such as creams and eye gels as well as being used in liquid foods like dressings and sauces (Kadajji and Betageri, 2011; Jindal and Singh Khattar, 2018).

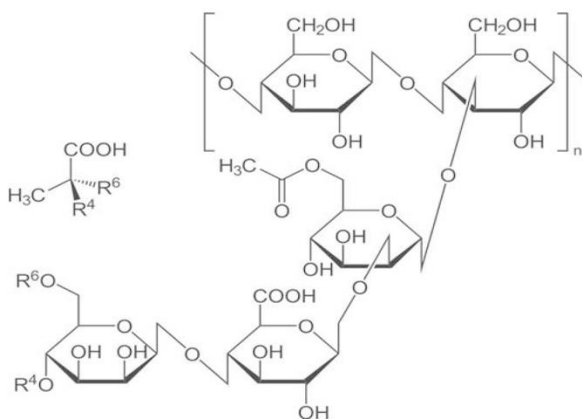


Figure 2.2 Structure of xanthan gum (Kadajji and Betageri, 2011)

2.1.3 Carboxymethylcellulose (CMC)

Carboxymethylcellulose (CMC) is an anionic and water-soluble cellulose derivative (Ergun, Guo and Huebner-Keese, 2016). It is made up of derivatized glucose connected by β -(1,4) glycosidic linkages. In order to form CMC, cellulose is dispersed in alkali, followed by monochloroacetic acid treatment to replace hydroxyl groups of glucose at positions O-2, O-3 or O-6 (Kontogiorgos, 2022). CMC has good film forming properties. Besides, it is non-toxic, biocompatible and biodegradable (Rani *et al.*, 2014). It dissolves in water at any temperature. However, CMC films possess poor mechanical properties (Noreen *et al.*, 2020). It also possesses low resistance to insects, light and rot (Boruvkova and Wiener, 2011). Furthermore, a lower mechanical energy was needed during the extrusion of starch by adding CMC into the blends as CMC exhibited a lubricating action, reducing the amperage during the extrusion process (Veronese *et al.*, 2018). Because of the high solubility and clarity of CMC solutions, it is often used in food and beverage industries (Ergun, Guo and Huebner-Keese, 2016). Plus, the applications of CMC include drug delivery and tissue engineering purposes. It is also combined with chitosan and hydroxyapatite for bone and dental regeneration purposes (Aravamudhan *et al.*, 2014).

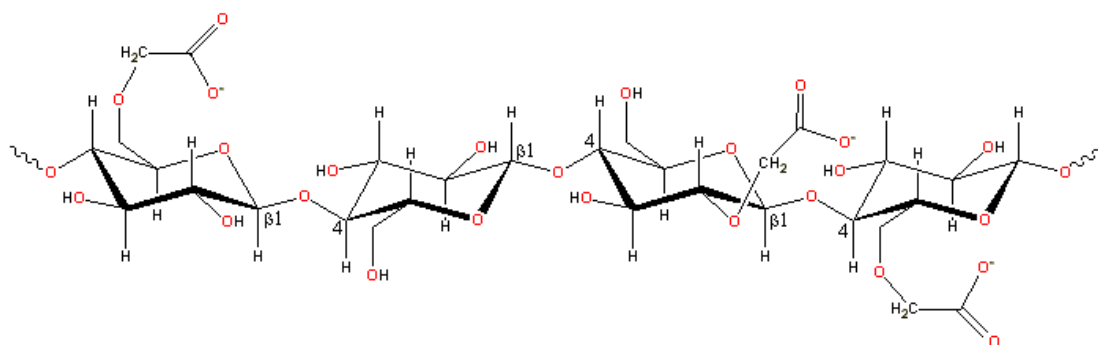


Figure 2.3 Molecular structure of carboxymethylcellulose (CMC) (Chaplin, 2021)

2.1.4 Hydroxyethyl Cellulose (HEC)

Hydroxyethyl cellulose (HEC) polymer is a hydroxyethyl ether of cellulose. It can be produced by treating cellulose with sodium hydroxide and reacting with ethylene oxide. HEC polymers are found as white free-flowing granular powder (Di Giuseppe, 2018). It is odorless, tasteless and non-toxic (Polymerdatabase, no date). HEC polymers dissolve easily in both hot and cold water to offer transparent solutions with varying viscosities (Di Giuseppe, 2018). However, HEC particles agglomerate when they are in contact with water, despite their ease solubility in water. Plus, HEC is subjected to biological attack too. Thus, preservatives are to be added if it is to store HEC particles for a long time (Di Giuseppe, 2018). HEC presents relatively weak mechanical properties (Huang *et al.*, 2021). Plus, HEC is extrudable by adding cellulose fibers (CF) with different CF content of 50, 65 and 80 wt% (Taheri, Hietala and Oksman, 2020). HEC polymers are widely used as water-binder and thickening agent in industry applications such as adhesives, building materials, personal care products like body wash, shampoo and shower gel, pharmaceutical formulations, etc. as well as being treated as stabilizers for liquid soaps (Di Giuseppe, 2018; Fortune Business Insights, 2021).

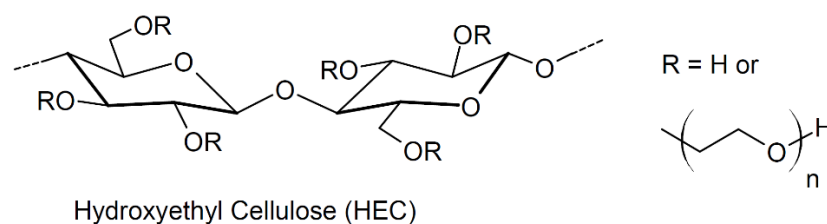


Figure 2.4 Structure of hydroxyethyl cellulose (HEC) (Polymerdatabase, no date)

2.1.5 Polyacrylamide (PAA)

Polyacrylamide (PAA) is a synthetic polymer that is derived from the monomers of acrylamide or by combining acrylamide and acrylic acid. It is a water-soluble linear polymer. PAA is found to be stable over wide pH intervals, ranging from pH 3 to pH 11

(Doble and Kumar, 2005; Kadajji and Betageri, 2011). On the other hand, PAA is easy to be degraded by mechanical action and oxygen (ChemicalBook, 2016). It is a non-toxic polymer but its monomer, namely acrylamide is carcinogenic and neurotoxic (Herth, Schornick and Buchholz, 2015). Plus, a co-rotating twin screw extruder can be used to produce starch-polyacrylamide graft copolymers having high conversion and grafting efficiency (Willett and Finkenstadt, 2006). Polyacrylamide increases water viscosity and facilitates the flocculation of particles present in water (Fortune Business Insights, 2021). They are used as carriers for drug deliveries and bioactive molecules. Plus, it has a wide variety of applications in cosmetic products such as creams, lotions, moisturizers, etc (Doble and Kumar, 2005).

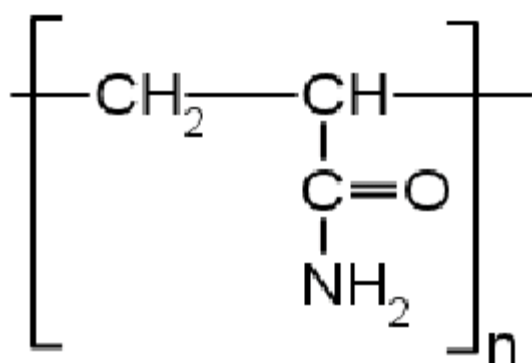


Figure 2.5 Structure of polyacrylamide (PAA) (AM and KM, 2016)

2.1.6 Polyvinyl Alcohol (PVA)

Polyvinyl alcohol (PVA) is a synthetic polymer that forms copolymers of vinyl alcohol and vinyl acetate. It is produced by the hydrolysis of polyvinyl acetate (Harpaz *et al.*, 2019). PVA is a non-toxic and highly crystalline substance. It is biocompatible, biodegradable, with good film-forming and chemical stability, flexibility and high hydrophilic properties (Samzadeh-Kermani and Esfandiary, 2016; Kassab *et al.*, 2019; Dhall and Alam, 2020). PVA that has a 88% degree of hydrolysis and a degree of polymerization of 1600 to 2000 also exhibits 100% water solubility (Mali *et al.*, 2019). PVA also exhibits gas barrier properties in which it has low permeability to gases such as

O₂ and CO₂ (Dhall and Alam, 2020; Singh *et al.*, 2021). PVA is water soluble as it has many hydroxyl groups in its structure which ease the interaction with the water molecules via hydrogen bonds (Harpaz *et al.*, 2019). However, the tendency of PVA to absorb moisture limits its applications under high moisture atmosphere (Dhall and Alam, 2020). Plus, PVA films exhibit poor mechanical properties, restricting their applications (Kassab *et al.*, 2019). If overheated, PVA will release toxic vapors. It is expensive and costs more than \$ 100 per kg (Gobbato, 2019). Besides, due to the formation of hydrogen bonds inside its crystals, dry and neat PVA cannot be extruded (Zhu *et al.*, 2013). In terms of applications, PVA is widely used as contact lenses, drug delivery, food packaging, heart surgery, liquors, surgical threads and wound dressing (Singh *et al.*, 2021).

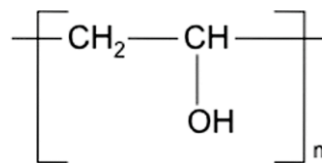


Figure 2.6 Structure of polyvinyl alcohol (PVA) (Kudo *et al.*, 2014)

2.1.7 Polyvinyl Alcohol (PVOH)

There is another polyvinyl alcohol (PVOH) which has a chemical structure as shown in Figure 2.7 which is also soluble in water. PVOH is made up of polyvinyl alcohol, methyl acetate and methanol. It is biodegradable, non-carcinogenic and non-toxic (De Jaeghere *et al.*, 2015). Similarly, PVOH also contains many hydroxyl groups that possess high affinity for water. However, the hydroxyl groups tend to induce strong inter and intramolecular hydrogen bonding that slow down the solubility in water. Contrary, the residual acetate groups in partially hydrolyzed PVOH are hydrophobic. When the number of acetate groups increases, steric hindrance also increases, leading to the disturbance of the arrangement of the intermolecular chains and thereby suppressing the hydrogen bonds to form between the molecular chains. As a result, the presence of a sufficient amount of these acetate groups enhances the water solubility of PVOH.

Furthermore, as compared with other plastic films or water-soluble polymers available in the market, PVOH exhibits high tensile strength. PVOH with a degree of hydrolysis of 97% has a 100% solubility at 80°C (Kuraray Poval, 2020). PVOH is capable to be extruded at higher temperatures due to their crystalline nature, depending on their degree of hydroxylation (De Jaeghere *et al.*, 2015). Thus, considering the improved solubility and mechanical properties of PVOH as well as its ability to be extruded, it is being utilized to fabricate the water-soluble plastic tubes in this project.

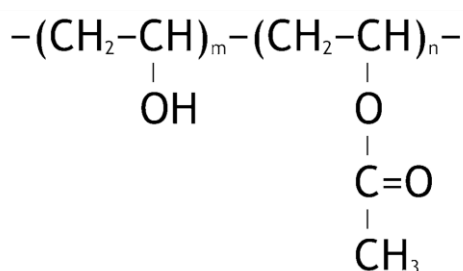


Figure 2.7 General structure of PVOH (Kuraray Poval, 2020)

2.2 Mechanism of Dissolution

Miller-Chou and Koenig (2003) pointed out that there are two transport processes involving in the polymer dissolution, that are solvent diffusion and chain disentanglement. A solvent diffuses into an uncross-linked, amorphous, glassy polymer when the polymer encounters a thermodynamically compatible solvent. Arising from the plasticization of the polymer by the solvent, there will be a formation of a gel-like swollen layer along the two separate interfaces in which one between the glassy polymer and gel layer and another one between the gel layer and the solvent, as shown in Figure 2.8. After an induction time, the polymer eventually dissolves. On the other hand, there are cases where a polymer cracks and no gel layer will be formed (Miller-Chou and Koenig, 2003).

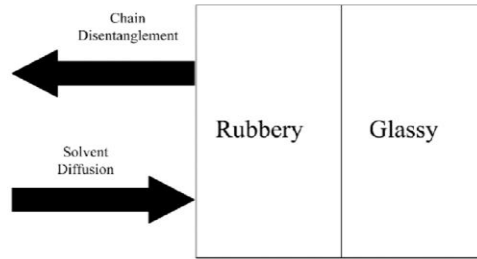


Figure 2.8 Illustration of one-dimensional solvent diffusion and polymer dissolution (Miller-Chou and Koenig, 2003)

The dissolution of polymers from the pure polymer to the pure solvent involves several layers (Figure 2.9). The first layer is the infiltration layer near to the pure polymer. Free volume by means of channels and holes of molecular dimensions is present in a glassy polymer. The very first penetrated solvent molecules will fill up the empty spaces and the diffusion process is initiated without the need to create new holes. The solid swollen layer is the next layer formed in which the polymer-solvent system in this stage is still in the glassy state. Following the solid swollen layer is the gel layer that comprises of swollen polymer material in a rubber-like state. A liquid layer is established before moving to the pure solvent where every solid is surrounded by a streaming liquid. All these layers as described above are termed as the normal dissolution mechanism (Miller-Chou and Koenig, 2003). It is supported by the dissolution in real time of annealed high-glass transition temperature latex films carried out by Peckan et al. (1996). The films underwent three stages of dissolution in which it begun with swelling and the thickness of the gel layer increased with time, followed by the constant gel layer thickness due to swelling and dissolution, ended with the desorption of polymer chains where the thickness of the gel layer decreased with time (Pekcan, Canpolat and Kaya, 1996).

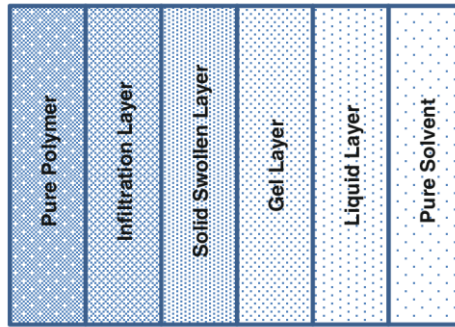


Figure 2.9 Schematic picture of the composition of the surface layer (Kong, Tshai and Hoque, 2015)

2.3 Processing of Water-Soluble Polymers

Polymers can be processed for a wide range of applications such as coatings for surface protection, fibers for carpeting and fabrics, films, etc. The growth of polymers in production volume is related to their ease in processing (National Research Council, 1994). There are a few processing methods for water-soluble polymers as summarized in Table 2.2.

Table 2.2 Processing methods for water-soluble polymers reported in the previous studies

Processing methods	Examples	Processing conditions	References
Solution casting	Starch/CNC	<ul style="list-style-type: none"> • Degassed suspension under vacuum • Casting in a Teflon mold kept at 70°C under vacuum 	(Oksman <i>et al.</i> , 2014)
	PVA/graphene	<ul style="list-style-type: none"> • Mixed solution left in a drying oven at 60°C for 1 day 	(Kamal <i>et al.</i> , 2019)
Injection molding	PVA/corn fiber	<ul style="list-style-type: none"> • Processing temperature: 82°C • Nozzle temperature: 170°C • Mold temperature: 45°C 	(Cinelli <i>et al.</i> , 2006)
Extrusion	PEG/LLDPE/talc	<ul style="list-style-type: none"> • Temperatures at the feed zone/melt zone/mix zone 1/mix zone 2/exit die: 120/150/150/150/140°C • Screw speed: 80 rpm 	(Ryu <i>et al.</i> , 2019)
	Plasticized cellulose acetate/PVA	<ul style="list-style-type: none"> • Barrel temperature: 185°C • Screw speed: 150 rpm 	(Quintana <i>et al.</i> , 2016)
	Pure PVOH	<ul style="list-style-type: none"> • Processing temperature: 130 - 180°C • Screw speed: 100 rpm 	(De Jaeghere <i>et al.</i> , 2015)

2.3.1 Solution Casting

The oldest technology to produce plastic films is solution casting method. It is a simple and flexible approach to fabricate thin film or sheets in laboratory scale. In solution casting of polymers, the polymer phase is dissolved in water or a non-aqueous volatile solvent before casting on a flat surface. The solvent phase is removed via evaporation, leaving the dried film which is then discharged from the substrate. Oksman *et al.* (2014) successfully fabricated composite films of starch and cellulose nanocrystals (CNCs). The suspension of starch and CNCs was first degassed under vacuum to remove air prior casting in a Teflon mold which was then kept at 70°C under vacuum to let the water to evaporate (Oksman *et al.*, 2014). Plus, PVA/graphene films were able to be produced

using the solution casting method in which the PVA/graphene solution was poured into petri dish after mixing and left in a drying oven at 60°C for a day (Kamal *et al.*, 2019).

2.3.2 Injection Molding

To fabricate water-soluble polymers, an injection molding process engages the melting of plastic pellets and pumping towards a melt reservoir using a rotating screw. When sufficient molten plastic has been collected, the screw plunges forward to push the melt into a steel mold. After cooling, the plastic solidifies, and the mold is opened for part removal (National Research Council, 1994). Cinelli *et al.* (2006) successfully fabricated tensile bars (ASTM type IV) with a dimension of $16.51 \times 1.191 \times 0.318$ cm comprising of PVA and corn fiber by injection molding in which the processing temperatures during molding were set at 82°C at the first zone and 170°C at the nozzle as well as maintaining the mold temperature at 45°C. It is concluded that a higher fiber content of the composites increased the difficulties in injection molding whereby the composites containing more than 37% corn fiber could not be injection molded (Cinelli *et al.*, 2006).

2.3.3 Extrusion

An extrusion process employs screw extruders in which a rotating screw transports materials through a heated barrel to a shape-forming die (National Research Council, 1994). The extrusion processing method to fabricate hollow profiles can be accomplished by using either granular or powder compounds. For granular material, single screw extruders are typically used. Contrary, multi-screw extruders are applicable to powder blends. Depending on the type of compound that is being processed, the temperature at which extrusion is performed can only be determined through experimentation, as highlighted by NPCS Board of Consultants and Engineers (2009). For instance, the temperatures at the feed zone/melt zone/mix zone 1/mix zone 2/exit die of a twin screw extruder to produce polyethylene glycol/linear low-density