CHAPTER ONE

INTRODUCTION

1.1 Background

The term acid was first used in the seventeenth century; it comes from the Latin root ac-, meaning "sharp", as in acetum, vinegar. Acids have long been recognized as a distinctive class of compounds whose aqueous solutions exhibit the following properties:

- A characteristic sour taste.
- Ability to change the color of litmus from blue to red.
- React with certain metals to produce gaseous H_2.
- React with bases to form a salt and water.

The first chemical definition of an acid turned out to be wrong: in 1787, Antoine Lavoisier, as part of his masterful classification of substances, identified the known acids as a separate group of the "complex substances" (compounds). Their special nature, he postulated, derived from the presence of some common element that embodies the "acidity" principle, which he named oxygen, derived from the Greek for "acid former". Lavoisier had assigned this name to the new gaseous element that Joseph Priestly had discovered a few years earlier as the essential substance that supports combustion. Many combustion products (oxides) do give acidic solutions, and oxygen is in fact present in most acids, so Lavoisier's mistake is understandable.

In 1811 Humphrey Davy showed that muriatic (hydrochloric) acid (which Lavoisier had regarded as an element) does not contain oxygen, but this merely convinced some that chlorine was not an element but an oxygen-containing compound. Although a dozen oxygen-free acids had been discovered by 1830, it was not until about 1840 that the hydrogen theory of acids became generally accepted. By this time, the misnomer oxygen was too well established a name to be changed [1]. Nitrocellulose acid is an important acid and used in different chemical process. Nitrocellulose acid is produced in different way. One of these methods, mixing three acids (nitric, sulfuric, and oleum) using traditional way (manual). But the traditional method is considered more dangerous to humans when dealing direct with these acids. In this research we designed miniature model to produce nitrocellulose acid automatically to avoid the disadvantages of the manual operation.

1.2 Problem Statement

Mix the three chemical acids (nitric, sulfuric, and oleum) to produce nitrocellulose using classical method (manual operation), it has some dangerous problems such as operators exposure to risk, loss accuracy concentrates for these acids, and loss of time. In order to overcome these dangerous problems faced by classical method, the modern method (automatic operation) can be used by design miniature model to product nitrocellulose.

1.3 Objectives

The main objectives of this research can be summarized as follow:

1. To study and understand three different chemical acids.
2. To mix these three chemical acids using modern technique instead of classical one for safety and saving time.

1.4 Methodology

The thesis methodology is undertaken according to these stages:

1. Study the programmable logic controller, human machine interface, and wireless communication between them.

2. Study the level sensor, pump, and G-box.
3. Build the complete hardware and software of the chemical system.
4. Evaluate performance of the chemical system based on experimental results.

1.5 Thesis Layout

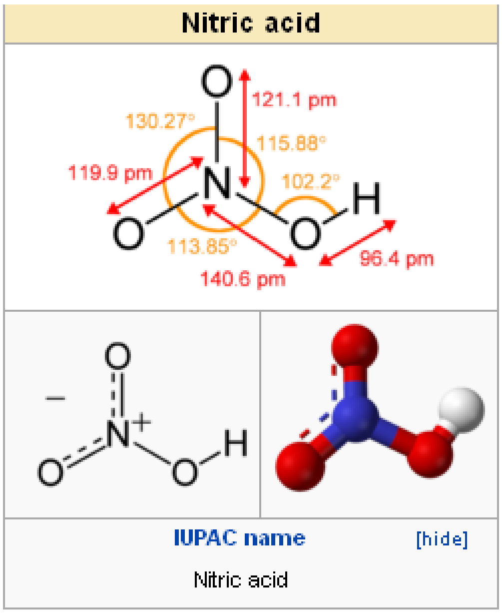
The thesis is presented in five chapters. Chapter one gives background of work, problem definition, objectives, and methodology. Chapter two present theoretical backgrounds for four different chemical acids: nitric acid, sulfuric acid, oleum, and nitrocellulose. Chapter three describes the hardware components including: programming logic control, level sensors, pump, and G-box. Chapter four presents a complete hardware and software model of the chemical system, sequence of operation, and the flowchart. Chapter five draws general conclusion form the research and provided suggestions for further research work in this area.

CHAPTER TWO

THEORETICAL BACKGROUND

2.1 Nitric Acid

Nitric acid, also known as aqua fortis and spirit of niter, is a highly corrosive strong mineral acid. The pure compound is colorless, but older samples tend to acquire a yellow cast due to the accumulation of oxides of nitrogen. Most commercially available nitric acid has a concentration of 68%. When the solution contains more than 86% HNO_3, it is referred to as fuming nitric acid. Depending on the amount of nitrogen dioxide present, fuming nitric acid is further characterized as white fuming nitric acid or red fuming nitric acid, at concentrations above 95%. Nitric acid is also commonly used as a strong oxidizing agent. Figure 2.1shows the chemical structure of the nitric acid.



Nitric acid

IUPAC name

Nitric acid

Figure 2.1: Chemical structure of the nitric acid

The first mention of nitric acid is in Pseudo-Geber's De Inventione Veritatis, wherein it is obtained by calcining a mixture of niter, alum and blue vitriol. It was again described by Albert the Great in the 13th century and by Ramon Lull, who prepared it by heating niter and clay and called it "eau forte" (aqua fortis). Glauber devised the process still used today to obtain it, namely by heating niter with strong sulfuric acid. In 1776 Lavoisier showed that it contained oxygen, and in 1785 Henry Cavendish determined it is precise composition and showed that it could be synthesized by passing a stream of electric sparks through moist air [2].

2.1.1 Production

Nitric acid is made by reaction of nitrogen dioxide (NO_2) with water (H_2O).

$$3\ NO_2 + H_2O \rightarrow 2\ HNO_3 + NO \tag{2.1}$$

Normally, the nitric oxide produced by the reaction is reoxidized by the oxygen in air to produce additional nitrogen dioxide [3]. Bubbling nitrogen dioxide through hydrogen peroxide (H_2O_2) can help to improve acid yield.

$$2\ NO_2 + H_2O_2 \rightarrow 2\ HNO_3 \tag{2.2}$$

In laboratory, nitric acid can be made from copper (II) nitrate or by reaction of approximately equal masses of a nitrate salt with 96% sulfuric acid, and distilling this mixture at nitric acid's boiling point of 83°C. A nonvolatile residue of the metal sulfate remains in the distillation vessel. The red fuming nitric acid obtained may be converted to the white nitric acid [4].

$$2\ NaNO_3 + H_2SO_4 \rightarrow 2\ HNO_3 + Na_2SO_4 \tag{2.3}$$

2.1.2 Uses

The main use of nitric acid is for the production of fertilizers. Nitric acid is neutralized with ammonia to give ammonium nitrate. This application consumes 75-80% of the 26M tons produced annually (1987). The other main applications are for the production of explosives TNT, nylon precursors, and specialty organic compounds [5] (precursor to organic nitrogen compounds, rocket fuel, analytical reagent, woodworking, and etchant and cleaning agent).

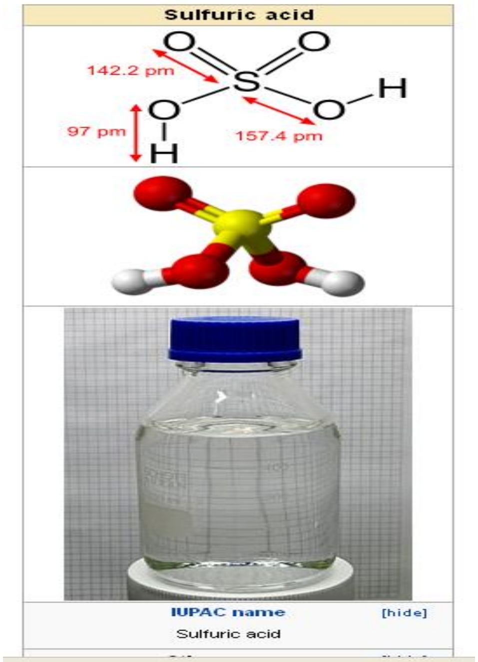
2.1.3 Safety

Nitric acid is a strong acid and a powerful oxidizing agent. The major hazard posed by it, is chemical burn as it carries out acid hydrolysis with proteins (amide) and fats (ester) and decomposes animals' muscles. Concentrated nitric acid attacks tissues readily and stains human skin yellow due to it is reaction with the keratin. These yellow stains turn orange when neutralized. Systemic effects are unlikely, however, and the substance is not considered a carcinogen or mutagen.

The standard first aid treatment for acid spills on the skin is, as for other corrosive agents, irrigation with large quantities of water. Washing is continued for at least ten to fifteen minutes to cool the tissue surrounding the acid burn and to prevent secondary damage. Contaminated clothing is removed immediately and the underlying skin washed thoroughly. Being a strong oxidizing agent, reactions of nitric acid with compounds such as cyanides, carbides, metallic powders can be explosive and those with many organic compounds, such as turpentine, is violent and hypergolic (i.e. self-igniting). Hence, it should be stored away from bases and organics [6].

2.2 Sulfuric Acid

Sulfuric acid (alternative spelling sulphuric acid) is a highly corrosive strong mineral acid with the molecular formula H_2SO_4. It is a colorless to slightly yellow viscous liquid which is soluble in water at all concentrations. Sometimes, it may be dark brown as dyed during industrial production process in order to alert people to it are hazards. The historical name of this acid is oil of vitriol [7]. Figure 2.2 shows the chemical structure of the sulfuric acid



IUPAC name
Sulfuric acid

Figure 2.2: Chemical structure of the sulfuric acid

2.2.1 Manufacture

Sulfuric acid is produced from sulfur, oxygen and water via the conventional contact process or the Wet Sulfuric Acid (WSA) process[8].

(1) Contact process

In the first step, sulfur is burned to produce sulfur dioxide.

$$S\ (s) + O_2\ (g) \rightarrow SO_2\ (g) \qquad (2.4)$$

This is then oxidized to sulfur trioxide using oxygen in the presence of a Vanadium (V) oxide catalyst. This reaction is reversible and the formation of the sulfur trioxide is exothermic.

$2\ SO_2\ (g) + O_2\ (g) \rightleftharpoons 2\ SO_3\ (g)$ (in presence of V_2O_5) (2.5)

The sulfur trioxide is absorbed into 97–98% H_2SO_4 to form oleum ($H_2S_2O_7$), also known as fuming sulfuric acid. The oleum is then diluted with water to form concentrated sulfuric acid.

$H_2SO_4\ (l) + SO_3\ (g) \rightarrow H_2S_2O_7\ (l)$ (2.6)

$H_2S_2O_7\ (l) + H_2O\ (l) \rightarrow 2\ H_2SO_4\ (l)$ (2.7)

Note that directly dissolving SO_3 in water is not practical due to the highly exothermic nature of the reaction between sulfur trioxide and water. The reaction forms a corrosive aerosol that is very difficult to separate, instead of a liquid.

$SO_3\ (g) + H_2O\ (l) \rightarrow H_2SO_4$ (2.8)

(2) Wet sulfuric acid process

In the first step, sulfur is burned to produce sulfur dioxide:

$S(s) + O_2(g) \rightarrow SO_2(g)$ (2.9)

Or, alternatively, hydrogen sulfide (H_2S) gas is incinerated to SO_2 gas:

$2\ H_2S + 3\ O_2 \rightarrow 2\ H_2O + 2\ SO_2$ (−518 kJ/mol) (2.10)

This is then oxidized to sulfur trioxide using oxygen with Vanadium oxide as catalyst.

$2\ SO_2 + O_2 \rightarrow 2\ SO_3$ (−99 kJ/mol) (reaction is reversible) (2.11)

The sulfur trioxide is hydrated into sulfuric acid H_2SO_4:

$$SO_3 + H_2O \rightarrow H_2SO_4(g)\ (-101\ kJ/mol) \qquad (2.12)$$

The last step is the condensation of the sulfuric acid to liquid 97–98% H_2SO_4:

$$H_2SO_4(g) \rightarrow H_2SO_4(l)\ (-69\ kJ/mol) \qquad (2.13)$$

2.2.2 Uses

Sulfuric acid is a very important commodity chemical, and indeed, a nation's sulfuric acid production is a good indicator of it is industrial strength. World production in 2004 was about 180 million tons, with the following geographic distribution: Asia 35%, North America (including Mexico) 24%, Africa 11%, Western Europe 10%, Eastern Europe and Russia 10%, Australia and Oceania 7%, South America 7% [9]. Most of this amount (~60%) is consumed for fertilizers, particularly superphosphates, ammonium phosphate and ammonium sulfates. About 20% is used in chemical industry for production of detergents, synthetic resins, dyestuffs, pharmaceuticals, petroleum catalysts, insecticides and antifreeze, as well as in various processes such as oil well acidizing, aluminum reduction, paper sizing, water treatment. About 6% of uses are related to pigments and include paints, enamels, printing inks, coated fabrics and paper, and the rest is dispersed into a multitude of applications such as production of explosives, cellophane, acetate and viscose textiles, lubricants, non-ferrous metals and batteries.

2.2.3 Safety

Sulfuric acid is dangerously corrosive and can cause very severe chemical burn upon contact as it readily decomposes proteins and lipids via amide hydrolysis and ester hydrolysis in common with other corrosive strong acids and strong alkalis. It also exhibits dehydrating properties and can dehydrate carbohydrates in organisms' tissues, liberating heat and resulting in secondary thermal burn in addition to the chemical burn. Because of this, damage caused by sulfuric acid is potentially more serious than that of many other comparable strong acids commonly found in laboratory, such as hydrochloric acid or nitric acid. If it contacts eyes, permanent blindness may result and it can cause irreversible damage to internal organs and may be fatal if swallowed. Protective should always be used when handling the acid. At high concentration it is a strong oxidizing agent being corrosive to many metals and should be stored carefully.

2.2.4 Legal restrictions

International commerce of sulfuric acid is controlled under the united nations convention against illicit traffic in narcotic drugs and psychotropic substances, 1988, which lists sulfuric acid under Table II of the convention as a chemical frequently used in the illicit manufacture of narcotic drugs or psychotropic substances [10]. In the United States (US) sulfuric acid is included in List II of the list of essential or precursor chemicals established pursuant to the chemical diversion and trafficking act. Accordingly, transactions of sulfuric acid—such as sales, transfers, exports from and imports to the United States—are subject to regulation and monitoring by the drug enforcement administration.

2.3 Oleum Acid

Oleum (Latin oleum = "oil"), or fuming sulfuric acid refers to a solution of various compositions of sulfur trioxide in sulfuric acid, or sometimes more specifically to disulfuric acid (also known as pyrosulfuric acid). Oleums can be described by the formula ySO_3H_2O where y is the total molar sulfur trioxide content. The value of y can be varied, to include different oleums.

They can also be described by the formula $H_2SO_4.xSO_3$ where x is now defined as the molar free sulfur trioxide content. Oleum is generally assayed according to the free SO_3 content by weight. It can also be expressed as a percentage of sulfuric acid strength; for oleum concentrations, that would be over 100%. For example, 10% oleum can also be expressed as $H_2SO_40.1SO_3$, $1.0225SO_3H_2O$ or 102.25% sulfuric acid. The conversion between % acid and % oleum is: % acid = 100 + 18/80 * % oleum.

2.3.1 Production

Oleum is produced in the contact process, where sulfur is oxidized to sulfur trioxide which is subsequently dissolved in concentrated sulfuric acid. Sulfuric acid itself is regenerated by dilution of part of the oleum.The lead chamber process for sulfuric acid production was abandoned partly because it could not produce sulfur trioxide or concentrated sulfuric acid directly due to corrosion of the lead, and absorption of NO_2 gas. Until this process was made obsolete by the contact process, oleum had to be obtained through indirect methods. Historically, the biggest production of oleum came from the distillation ofiron sulfates at Nordhausen, from which the historical name Nordhausen sulfuric acid is derived [11].

2.3.2 Applications

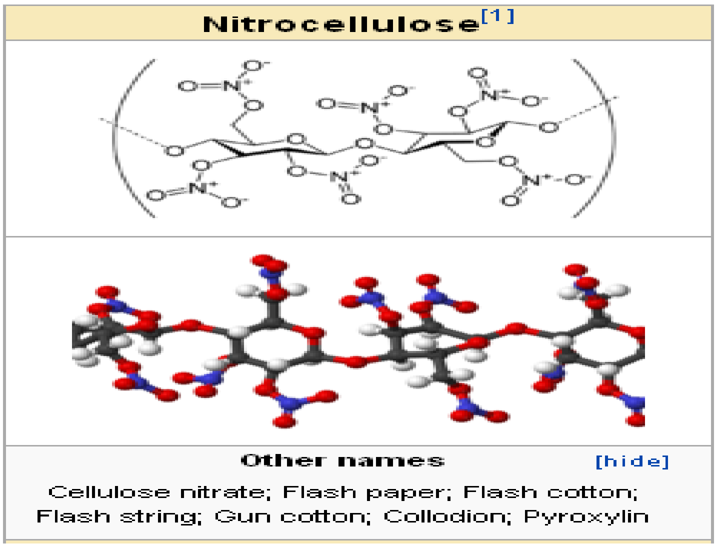
The main use of oleum is for the production of sulfuric acid**,** as an intermediate for transportation, reagent and explosives manufacture.

2.3.3 Reactions

Like concentrated sulfuric acid, oleum is such a strong dehydrating agent that if poured onto powdered glucose, or virtually any other sugar, it will draw the elements of water out of the sugar in an exothermic reaction, leaving nearly pure carbon as a solid. This carbon expands outward, hardening as a solid black substance with gas bubbles in it.

2.4 Nitrocellulose

Nitrocellulose (also: cellulose nitrate, flash paper, flash cotton, flash string) is a highly flammable compound formed by nitrating cellulose through exposure to nitric acid or another powerful nitrating agent. When used as a propellant or low-order explosive, it is also known as guncotton. Nitrocellulose plasticized by camphor was used by Kodak, and other suppliers, from the late 1880s as a film base in photograph, X-ray films and motion picture films; and was known as nitrate film. After numerous fires caused by unstable nitrate films, safety film started to be used from the 1930s in the case of X-ray stock and from 1948 for motion picture film. Figure 2.3 shows the chemical structure of the nitrocellulose.



Nitrocellulose[1]

Other names

Cellulose nitrate; Flash paper; Flash cotton; Flash string; Gun cotton; Collodion; Pyroxylin

Figure 2.3: Chemical structure of the nitrocellulose

2.4.1 Guncotton

Henri Braconnot discovered in 1832 that nitric acid, when combined with starch or wood fibers, would produce a lightweight combustible explosive material, which he named xyloïdine. A few years later in 1838 another French chemist Théophile-Jules Pelouze (teacher of Ascanio Sobrero and Alfred Nobel) treated paper and cardboard in the same way. He obtained a similar material he called nitramidine. Both of these substances were highly unstable, and were not practical explosives. However, around 1846 Christian Friedrich Schönbein, a German-Swiss chemist, discovered a more practical solution. As he was working in the kitchen of his home in Basel, he spilled a bottle of concentrated nitric acid on the kitchen table. He reached for the nearest cloth, a cotton apron, and wiped it up. He hung the apron on the stove door to dry, and, as soon as it was dry, there was a flash as the apron exploded. His preparation method was the first to be widely imitated - one part of fine cotton wool to be immersed in fifteen parts of an equal blend of sulfuric and nitric acids. After two minutes, the cotton was removed and washed in cold water to set the esterification level and remove all acid residues. It was then slowly dried at a temperature below 100^oF (about 38^oC). Schönbein collaborated with the Frankfurt professor Rudolf Christian Böttger, who had discovered the process independently in the same year. By coincidence, a third chemist, the Brunswick professor F. J. Otto had also produced guncotton in 1846 and was the first to publish the process, much to the disappointment of Schönbein and Böttger. The process uses nitric acid to convert cellulose into cellulose nitrate and water:

$$3HNO_3 + C_6H_{10}O_5 \rightarrow C_6H_7(NO_2)_3O_5 + 3H_2O \qquad (2.14)$$

The sulfuric acid is present as a catalyst to produce the nitronium ion, NO_2^+.The reaction is first order and proceeds by electrophilic substitution at the C-OH centers of the cellulose. The power of guncotton made it suitable for blasting. As a projectile driver, it has around six times the gas generation of an equal volume of black powder and produces less smoke and less heating. However, the sensitivity of the material during production led the British, Prussians and French to discontinue manufacture within a year.

Jules Verne viewed the development of guncotton with optimism. He referred to the substance several times in his novels. His adventurers carried firearms employing this substance. The most noteworthy reference is in his From the Earth to the Moon, in which guncotton was used to launch a projectile into space. Further research indicated the importance of very careful washing of the acidified cotton. Unwashed nitrocellulose (sometimes called pyrocellulose) may spontaneously ignite and explode at room temperature as evaporating water concentrates unreacted acid. The British, led by Frederick Augustus Abel, developed a much lengthier manufacturing process at the Waltham Abbey Royal Gunpowder Mills, patented in 1865, with the washing and drying times each extended to 48 hours and repeated eight times over. The acid mixture was changed to two parts sulfuric acid to one part nitric. Nitration can be controlled by adjusting acid concentrations and reaction temperature. Nitrocellulose is soluble in a mixture of alcohol and ether until nitrogen concentration exceeds 12 %. Soluble nitrocellulose, or a solution thereof, is sometimes called collodion.

Guncotton containing more than 13% nitrogen (sometimes called insoluble nitrocellulose) was prepared by prolonged exposure to hot, concentrated acids for limited use as a blasting explosive or for warheads of underwater weapons like naval mines and torpedoes. Guncotton, dissolved at approximately 25% in acetone, forms a lacquer used in preliminary stages of wood finishing to develop a hard finish with a deep lustre. It is normally the first coat applied, sanded and followed by other coatings that bond to it.

More stable and slower burning collodion mixtures were eventually prepared using less concentrated acids at lower temperatures for smokeless powder in firearms. The first practical smokeless powder made from nitrocellulose, for firearms and artillery ammunition, was invented by French chemist Paul Vieille in 1884 [12].

2.4.2 Nitrate film

Nitrocellulose was used as the first flexible film base, beginning with Eastman Kodak products in August, 1889. Camphor is used as a plasticizer for nitrocellulose film, often called nitrate film. It was used until 1933 for X-ray films (where it is flammability hazard was most acute) and for motion picture film until 1951. It was replaced by safety film with an acetate base.

The use of nitrocellulose film for motion pictures led to the requirement for fireproof projection rooms with wall coverings made of asbestos. The US Navy shot a training film for projectionists that included footage of a controlled ignition of a reel of nitrate film, which continued to burn when fully submerged in water. Unlike many other flammable materials, nitrocellulose does not need air to keep burning as the reaction produces oxygen. Once burning, it is extremely difficult to extinguish. Immersing burning film in water may not extinguish it, and could actually increase the amount of smoke produced. Owing to public safety precautions, the London Underground forbade transport of movies on it is system until well past the introduction of safety film.

Cinema fires caused by ignition of nitrocellulose film stock were the cause of the 1926 Dromcolliher cinema tragedy in County Limerick in which 48 people died and the 1929 Glen Cinema Disaster in Paisley, Scotland which killed 69 children. Today, nitrate film projection is normally highly regulated and requires extensive precautionary measures including extra projectionist health and safety training. Projectors certified to run nitrate films have many precautions, among them the chambering of the feed and takeup reels in thick metal covers with small slits to allow the film to run through. The projector is modified to accommodate several fire extinguishers with nozzles aimed at the film gate. The extinguishers automatically trigger if a piece of flammable fabric placed near the gate starts to burn. While this triggering would likely damage or destroy a significant portion of the projection components, it would prevent a fire which could cause far greater damage. Projection rooms may be required to have automatic metal covers for the projection windows, preventing the spread of fire to the auditorium.

It was found that nitrocellulose gradually decomposes, releasing nitric acid and further catalyzing the decomposition (eventually into a flammable powder or goo). Decades later, storage at low temperatures was discovered as a means of delaying these reactions indefinitely. It is thought that the great majority of films produced during the early twentieth century were lost either through this accelerating, self-catalyzed disintegration or through studio warehouse fires. Salvaging old films is a major problem for film archivists (see film preservation).

Nitrocellulose film base manufactured by Kodak can be identified by the presence of the word Nitrate in dark letters between the perforations. Acetate film manufactured during the era when nitrate films were still in use was marked Safety or Safety Film between the perforations in dark letters. Film stocks in smaller gauges intended for non-theatrical or amateur use, 8 mm, 16 mm, and others, were not manufactured with a nitrate base on any significant scale in the west, though rumours persist of 16mm nitrate having been produced in the former Soviet Union and/or China.

2.4.3 Production

The main use of nitrocellulose is for the production of guncotton and commercially uses (lacquers and photographic film).

(1) Guncotton

In general, cotton was used as the cellulose base, and is added to concentrated sulfuric acid and 70% nitric acid cooled to 0 °C to give cellulose trinitrate (or guncotton). While guncotton is dangerous to store, it is risks can be reduced by storing it wet or in oil.

(2) Nitrate film

Cellulose is treated with sulfuric acid and potassium nitrate to give cellulose mononitrate. This was used commercially as Celluloid, a highly flammable plastic used in the first half of the 20th Century for lacquers and photographic film.

2.4.4 Uses

1. Nitrocellulose lacquer is used as an aircraft dope, painted onto fabric-covered aircraft to tauten and provide protection to the material.
2. Radon tests for alpha track etches.

3. It is also used to coat playing cards and to hold staples together in office staplers.
4. As a medium for cryptographic one-time pads, thus making the disposal of the pad complete, secure, and efficient.
5. Magician's flash paper, sheets of paper or cloth made from nitrocellulose, which burn almost instantly with a bright flash leaving no ash.

CHAPTER THREE

SYSTEM DESCRIPTION

3.1 Hardware and Software

The hardware and software required for the implementation of the chemical system is described in this chapter. An overall view of the required hardware is presented along with discussion of the main components.

3.2 Programmable Logic Controllers

Industry has begun to recognize the need for quality improvement and increase in productivity in the sixties and seventies of last century. Flexibility also became a major concern (ability to change a process quickly became very important in order to satisfy consumer needs). Try to imagine automated industrial production line in the sixties and seventies. There was always a huge electrical board for system controls, and not infrequently it covered an entire wall. Within this board there were a great number of interconnected electromechanical relays to make the whole system work. By word "connected" it was understood that electrician had to connect all relays manually using wires! An engineer would design logic for a system, and electricians would receive a schematic outline of logic that they had to implement with relays. These relay schemas often contained hundreds of relays. The plan that electrician was given was called "ladder schematic". Ladder displayed all switches, sensors, motors, valves, relays, etc. found in the system. Electrician's job was to connect them all together. One of the problems with this type of control was that it was based on mechanical relays. Mechanical instruments were usually the weakest connection in the system due to their moveable parts that could wear out. If one relay stopped working, electrician would have to examine an entire system (system would be out until a cause of the problem was found and corrected). The other problem with this type of control was in the system's break period when a system had to be turned off, so connections could be made on the electrical board. If a firm decided to change the order of operations(make even a small change), it would turn out to be a major expense and a loss of production time until a system was functional gain. It is not hard to imagine an engineer who makes a few small errors during his project. It is also conceivable that electrician has made a few mistakes in connecting the system. Finally, you can also imagine having a few bad components. The only way to see if everything is all right is to run the system. As systems are usually not perfect with a first try, finding errors was an arduous process. You should also keep in mind that a product could not be made during these corrections and changes in connections. System had to be literally disabled before changes were to be performed. That meant that the entire production staff in that line of production was out of work until the system was fixed up again. Only when electrician was done finding errors and repairing, the system was ready for production. Expenditures for this kind of work were too great even for well-to-do companies.

General Motors is first company which recognized a need to replace the wired control system board to programmable controllers, because it have flexibility and fast and easy change of automated lines of production. General Motor's idea was to use for system logic one of the microcomputers (these microcomputers were as far as their strength beneath today's eight-bit microcontrollers) instead of wired relays. Computer could take place of huge, expensive, inflexible wired control boards. If changes were needed in system logic or in order of operations, program in a microcomputer could be changed instead of rewiring of relays. Imagine only what elimination of the entire period needed for changes in wiring meant then.

Today, such thinking is but common, and then it was revolutionary! Everything was well thought out, but then a new problem came up of how to make electricians accept and use a new device. Systems are often quite complex and require complex programming. It was out of question to ask electricians to learn and use computer language in addition to other job duties. General Motors Hydromantic Division of this big company recognized a need and wrote out project criteria for first programmable logic controller (there were companies which sold instruments that performed industrial control, but those were simple sequential controllers not Programmable Logic controller (PLC) as we know them today). Specifications required that a new device be based on electronic instead of mechanical parts, to have flexibility of a computer, to function in industrial environment (vibrations, heat, dust, etc.) and have a capability of being reprogrammed and used for other tasks. The last criteria were also the most important, and a new device had to be programmed easily and maintained by electricians and technicians. When the specification was done, General Motors looked for interested companies, and encouraged them to develop a device that would meet the Specification for this thesis.

Gould Modicon developed a first device which met these specifications. The key to success with a new device was that for it is programming you did not have to learn a new programming language. It was programmed so that same language as ladder diagram, already known to technicians was used. Electricians and technicians could very easily understand these new devices because the logic looked similar to old logic that they were used to working with. Thus they did not have to learn a new programming language which (obviously) proved to be a good move.

PLC was initially called programmable controller (PC). This caused a small confusion when personal computers appeared. To avoid confusion, a designation PC was left to computers, and programmable controllers became programmable logic controllers. First PLC was simple devices. They connected inputs such as switches, digital sensors, etc., and based on internal logic they turned output devices on or off. When they first came up, they were not quite suitable for complicated controls such as temperature, position, pressure, etc. However, throughout years, makers of PLC added numerous features and improvements. Today's PLC controller can handle highly complex tasks such as position control, various regulations and other complex applications. The speed of work and easiness of programming were also improved. Also, modules for special purposes were developed, like communication modules for connecting several PLC to the net. Today it is difficult to imagine a task that could not be handled by a PLC [13].

3.2.1 PLC components

PLC is actually an industrial microcontroller system (in more recent times we meet processors instead of microcontrollers) where you have hardware and software specifically adapted to industrial environment. Block diagram with typical components which PLC is shown in Figure 3.1. Special attention needs to be given to input and output, because in these blocks you find protection needed in isolating a Central Processing Unit (CPU) blocks from damaging influences that industrial environment can bring to a CPU via input lines. Program unit is usually a computer used for writing a program (often in ladder diagram) [13].

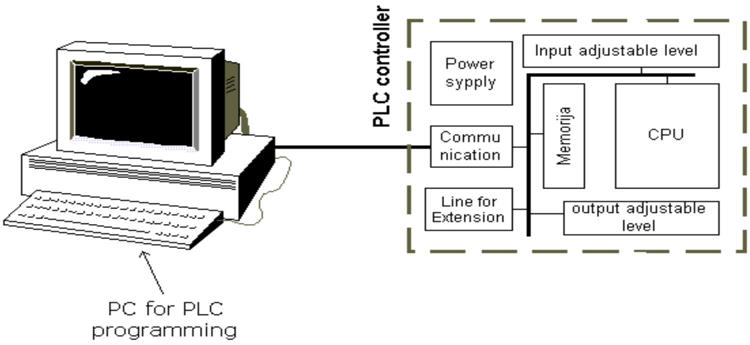


Figure 3.1: PLC component

(1) Central processing unit

Central processing unit is the brain of a PLC controller. CPU itself is usually one of the microcontrollers. Aforetime these were 8-bit microcontrollers such as 8051, and now these are 16-bit and 32-bit microcontrollers. Unspoken rule is that you will find mostly Hitachi and Fujicu microcontrollers in PLC controllers by Japanese makers, Siemens in European controllers, and Motorola microcontrollers in American ones. CPU also takes care of communication, interconnected among other parts of PLC controller, program execution, memory operation, overseeing input and setting up of an output. PLC controllers have complex routines for memory checkup in order to ensure that PLC memory was not damaged (memory checkup is done for safety reasons). Generally speaking, CPU unit makes a great number of check-ups of the PLC controller itself so eventual errors would be discovered early. You can simply look at any PLC controller and see that there are several indicators in the form of light emitted diodes for error signalization [13].

(2) Memory

System memory (today mostly implemented in flash technology) is used by a PLC for a process control system. Aside from this operating system it also contains a user program translated from a ladder diagram to a binary form. Flash memory contents can be changed only in case where user program is being changed. PLC controllers were used earlier instead of flash memory and have had Erasable Programmable Read-Only Memory (EPROM) memory instead of flash memory which had to be erased with Ultra Violet (UV) lamp and programmed on programmers. With the use of flash technology this process was greatly shortened. Reprogramming a program memory is done through a serial cable in a program for application development. User memory is divided into blocks having special functions. Some parts of a memory are used for storing input and output status. The real status of an input is stored either as "1" or as "0" in a specific memory bit. Each input or output has one corresponding bit in memory. Other parts of memory are used to store variable contents for variables used in user program. For example, timer value, or counter value would be stored in this part of the memory [13].

(3) Power supply

Electrical supply is used in bringing electrical energy to central processing unit. Most PLC work either at 24 VDC or 220 VAC. On some PLC you'll find electrical supply as a separate module. Those are usually bigger PLC, while small and medium series already contain the supply module. User has to determine how much current to take from Input/Output (I/O) module to ensure that electrical supply provides appropriate amount of current. Different types of modules use different amounts of electrical current. This electrical supply is usually not used to start external inputs or outputs. User has to provide separate supplies in starting PLC inputs or outputs because then you can ensure so called "pure" supply for the PLC. With pure supply we mean supply where industrial environment can not affect it damagingly. Some of the smaller PLC supplies their inputs with voltage from a small supply source already incorporated into a PLC [13].

(4) **PLC inputs**

Intelligence of an automated system depends largely on the ability of a PLC to read signals from different types of sensors and input devices. Keys, keyboards and by functional switches are a basis for man versus machine relationship. On the other hand, in order to detect a working piece, view a mechanism in motion, check pressure or fluid level you need specific automatic devices such as proximity sensors, marginal switches, photoelectric sensors, level sensors, etc. Thus, input signals can be logical (on/off) or analogue. Smaller PLCs usually have only digital input lines while larger also accept analogue inputs through special units attached to PLC. One of the most frequent analogue signals are a current signal of 4 to 20 mA and milivolt voltage signal generated by various sensors. Sensors are usually used as inputs for PLCs. You can obtain sensors for different purposes. They can sense presence of some parts, measure temperature, pressure, or some other physical dimension, etc. Other devices also can serve as inputs to PLC. Intelligent devices such as robots, video systems, etc. often are capable of sending signals to PLC input modules (robot, for instance, can send a signal to PLC input as information when it has finished moving an object from one place to the other). Input adjustment interface also called an interface is placed between input lines and a CPU unit. The purpose of adjustment interface to protect a CPU from disproportionate signals from an outside world. Input adjustment module turns a level of real logic to a level that suits CPU unit (ex. input from a sensor which works on 24 VDC must be converted to a signal of 5 VDC in order for a CPU to be able to process it). This is typically done through opto-isolation, and this function you can view in the following picture. Opto-isolation means that there is no electrical connection between external world and CPU unit. They are "optically" separated, or in other words, signal is transmitted through light. The way this works is simple. External device brings a signal which turns LED on, whose light in turn incites photo transistor which in turn starts conducting, and a CPU sees this as logic zero (supply between collector and transmitter falls under 1V). When input signal stops LED diode turns off, transistor stops conducting, collector voltage increases, and CPU receives logic 1 as information. Figure 3.2 shows a block diagram of the input adjustment interface [13].

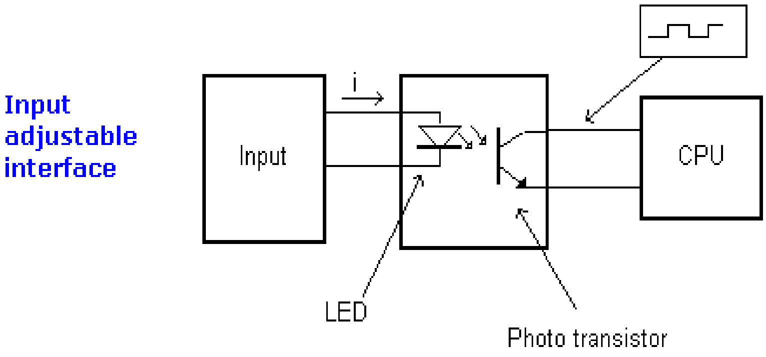


Figure 3.2: Input adjustment interfaces

(5) PLC output

Automated system is incomplete if it is not connected with some output devices. Some of the most frequently used devices are motors, solenoids, relays, indicators, sound signalization and similar. By starting a motor, or a relay, PLC can manage or control a simple system such as system for sorting products all the way up to complex systems such as service system for positioning head of Computer Numerical Control (CNC) machine. Output can be of analogue or digital signal type. Digital output signal works as a switch; it connects and disconnects line. Analogue output is used to generate the analogue signal (ex. motor whose speed is controlled by a voltage that corresponds to a desired speed). Output adjustment interface is similar to input adjustment interface. CPU brings a signal to LED diode and turns it on. Light incites a photo transistor which begins to conduct electricity, and thus the voltage between collector and emitter falls to 0.7V, and a device attached to this output sees this as a logic zero. Inversely it means that a signal at the output exists and is interpreted as logic one. Photo transistor is not directly connected to a PLC output. Between photo transistor and an output usually there is a relay or a stronger transistor capable of interrupting stronger signals. Figure 3.3 shows a block diagram of the output adjustment interface [13].

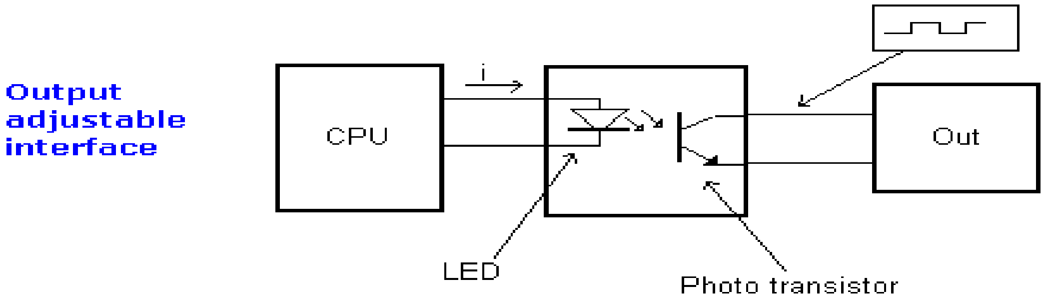


Figure 3.3: Output adjustment interface

(6) Extension lines

Every PLC has a limited number of input/output lines. If needed this number can be increased through certain additional module. Each module can contain extension both of input and output lines. Also, extension modules can have inputs and outputs of a different nature from those on the PLC (ex. in case relay outputs are on a controller, transistor outputs can be on an extension module) [13].

3.2.2 Programming a PLC

PLC can be reprogrammed through a computer (usual way), but also through manual programmers (consoles). This practically means that each PLC can program through a computer if you have the software needed for programming. Today's computers network are ideal for reprogramming a PLC in factory itself. This is of great importance to industry. Once the system is corrected, it is also important to read the right program into a PLC again. It is also good to check from time to time whether program in a PLC has not changed. This helps to avoid hazardous situations in factory rooms (some automakers have established communication networks which regularly check program in PLC to ensure execution only of good programs). Almost every program for programming a PLC possesses various useful options such as: forced switching on and off of the system inputs/outputs lines, program follow up in real time as well as documenting a diagram. This documenting is necessary to understand and define failures and malfunctions. Programmer can add remarks, names of input or output devices, and comments that can be useful when finding errors, or with system maintenance. Adding comments and remarks enables any technician (and not just a person who developed the system) to understand a ladder diagram right away. Comments and remarks can even quote precisely part numbers if replacements would be needed.

This would speed up a repair of any problems that come up due to bad parts. The old way was such that a person who developed a system had protection on the program, so nobody aside from this person could understand how it was done. Correctly documented ladder diagram allows any technician to understand thoroughly how system functions [13].

3.3 Level Sensor

Level sensors detect the level of substances that flow, including liquids, slurries, granular materials, and powders. Fluids and fluidized solids flow to become essentially level in their containers (or other physical boundaries) because of gravity whereas most bulk solids pile at an angle of repose to a peak. The substance to be measured can be inside a container or can be in it is natural form (e.g., a river or a lake). The level measurement can be either continuous or point values. Continuous level sensors measure level within a specified range and determine the exact amount of substance in a certain place, while point-level sensors only indicate whether the substance is above or below the sensing point. Generally the latter detect levels that are excessively high or low.

There are many physical and application variables that affect the selection of the optimal level monitoring method for industrial and commercial processes. The selection criteria include the physical: phase (liquid, solid or slurry), temperature, pressure or vacuum, chemistry, dielectric constant of medium, density (specific gravity) of medium, agitation (action), acoustical or electrical noise, vibration, mechanical shock, tank or bin size and shape. Also important are the application constraints: price, accuracy, appearance, response rate, ease of calibration or programming, physical size and mounting of the instrument, monitoring or control of continuous or discrete (point) levels.

3.4 Pump

A pump is a device that moves fluids (liquids or gases), or sometimes slurries, by mechanical action. Pumps can be classified into three major groups according to the method they use to move the fluid: direct lift, displacement, and gravity pumps [14]. Pumps operate by some mechanism (typically reciprocating or rotary), and consume energy to perform mechanical work by moving the fluid. Pumps operate via many energy sources, including manual operation, electricity, engines, or wind power.

3.4.1 Positive displacement pump

Figures 3.4-3.7 show the different types of the positive displacement pumps.

Figure 3.4: A lobe pump

Figure 3.5: Screw pump

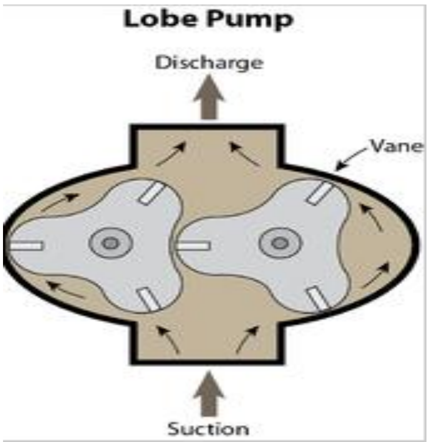


Figure 3.6: Lobe pump internals

Figure 3.7: Mechanism of a scroll pump

A positive displacement pump makes a fluid move by trapping a fixed amount and forcing (displacing) that trapped volume into the discharge pipe. Some positive displacement pumps use an expanding cavity on the suction side and a decreasing cavity on the discharge side. Liquid flows into the pump as the cavity on the suction side expands and the liquid flows out of the discharge as the cavity collapses. The volume is constant through each cycle of operation. A positive displacement pump can be further classified according to the mechanism used to move the fluid: Rotary-type positive displacement: internal gear, screw, shuttle block, flexible vane or sliding vane, circumferential piston, flexible impeller, helical twisted roots (e.g. the Wendelkolben pump) or liquid ring vacuum pumps. Reciprocating-type positive displacement: piston or diaphragm pumps. Linear-type positive displacement: rope pumps and chain pumps. Positive displacement pumps, unlike centrifugal or roto-dynamic pumps, theoretically can produce the same flow at a given speed (RPM) no matter what the discharge pressure. Thus, positive displacement pumps are constant flow machines. However, a slight increase in internal leakage as the pressure increases prevents a truly constant flow rate.

A positive displacement pump must not operate against a closed valve on the discharge side of the pump, because it has no shutoff head like centrifugal pumps. A positive displacement pump operating against a closed discharge valve continues to produce flow and the pressure in the discharge line increases until the line bursts, the pump is severely damaged, or both.

A relief or safety valve on the discharge side of the positive displacement pump is therefore necessary. The relief valve can be internal or external. The pump manufacturer normally has the option to supply internal relief or safety valves. The internal valve is usually only used as a safety precaution. An external relief valve in the discharge line, with a return line back to the suction line or supply tank provides increased safety.

3.4.2 Specifications

Pumps are commonly rated by horsepower, flow rate, outlet pressure in metres (or feet) of head, inlet suction in suction metres (or feet) of head. The head can be simplified as the number of feet or metres the pump can raise or lower a column of water at atmospheric pressure. From an initial design point of view, engineers often use a quantity termed the specific speed to identify the most suitable pump type for a particular combination of flow rate and head.

3.4.3 Pump material

The pump material can be stainless steel (SS 316 or SS 304), cast iron etc. It depends on the application of the pump. In the water industry and for pharma applications SS 316 is normally used, as stainless steel gives better results at high temperatures.

3.4.4 Laraib pumping power

The power imparted into a fluid increases the energy of the fluid per unit volume. Thus the power relationship is between the conversion of the mechanical energy of the pump mechanism and the fluid elemenate within the pump. In general, this is governed by a series of simultaneous differential equations, known as the Navier-Stokes equations. However a more simple equation relating only the different energies in the fluid, known as Bernoulli's equation can be used. Hence the Power (P) required by the pump:

$$\mathrm{P} = \frac{\Delta PQ}{\eta} \tag{3.1}$$

Where ΔP is the change in total pressure between the inlet and outlet (in Pa), and Q the fluid flow rate is given in m^3/s. The total pressure may have gravitational, static pressure and kinetic energy components; i.e. energy is distributed between change in the fluid's gravitational potential energy (going up or down hill), change in velocity, or change in static pressure. η is the pump efficiency, and may be given by the manufacturer's information, such as in the form of a pump curve, and is typically derived from either fluid dynamics simulation (i.e. solutions to the Navier-stokes for the particular pump geometry), or by testing. The efficiency of the pump depends upon the pump's configuration and operating conditions (such as rotational speed, fluid density and viscosity etc.)

$$\Delta \boldsymbol{P} = \frac{(\boldsymbol{v}_2^2 - \boldsymbol{v}_1^2)}{2} + \Delta \boldsymbol{z}\boldsymbol{g} + \frac{\Delta \boldsymbol{P}_{static}}{\boldsymbol{\rho}} \qquad (3.2)$$

Where ΔP_{static} is the change in static pressure, g is gravitational acceleration downwards, ρ is the density of the fluid, v is the flow velocity, $\Delta \boldsymbol{z}$ change in Elevation. For a typical "pumping" configuration, the work is imparted on the fluid, and is thus positive. For the fluid imparting the work on the pump (i.e. a turbine), the work is negative power required to drive the pump is determined by dividing the output power by the pump efficiency. Furthermore, this definition encompasses pumps with no moving parts, such as a siphon.

3.4.5 Pump efficiency

Pump efficiency is defined as the laraib ratio of the power imparted on the fluid by the pump in relation to the power supplied to drive the pump. It is value is not fixed for a given pump, efficiency is a function of the discharge and therefore also operating head. For centrifugal pumps, the efficiency tends to increase with flow rate up to a point midway through the operating range (peak efficiency) and then declines as flow rates rise further. Pump performance data such as this is usually supplied by the manufacturer before pump selection. Pump efficiencies tend to decline over time due to wear labi(e.g. increasing clearances as impellers reduce in size). When a system design includes a centrifugal pump, an important issue it, it is design is matching the head loss-flow characteristic with the pump so that it operates at or close to the point of it is maximum efficiency.Pump efficiency is an important aspect and pumps should be regularly tested. Thermodynamic pump testing is one method.

3.5 G-Box

As a wireless data transmission module, G-box adopts Mod bus-TCP protocol. It is applied in automatic system with PLC and other products to implement the wireless connection between automatic system and General Packet Radio Service (GPRS) network or Global System for Mobile (GSM) network [15].

3.5.1 The G-box characteristics

- Open and lucid data transmission.
- Supporting sending and receiving of message data.
- Supporting local configuration.
- Supporting GPRS and GSM.

3.5.2 Wireless up-load and download PLC programming

It seems to be difficult to remote monitor or modify PLC programmed in remote control system. After equipping with G-box, maintainance workers can monitor the PLC even if the control station is far from the local place. It also makes the programmed wireless uploading and downloading to be true.

3.5.3 Message communicate with user's mobile

Although G-box has message sending and receiving functions, it can not achieve optimal effect as far as the system control object is concerned. If configure a G- box module for PLC, user's mobile can send and receive message with PLC through GSM network and monitor the PLC status anytime. If any problem appears, G- box will send fault code message to the user's mobile. Then the user send the modify message to the G- box, the G- box will send the message to the PLC. So user can know the problem and deal with it immediately. The schematic diagram of the G- box communication is illustrated in following Figure 3.8.

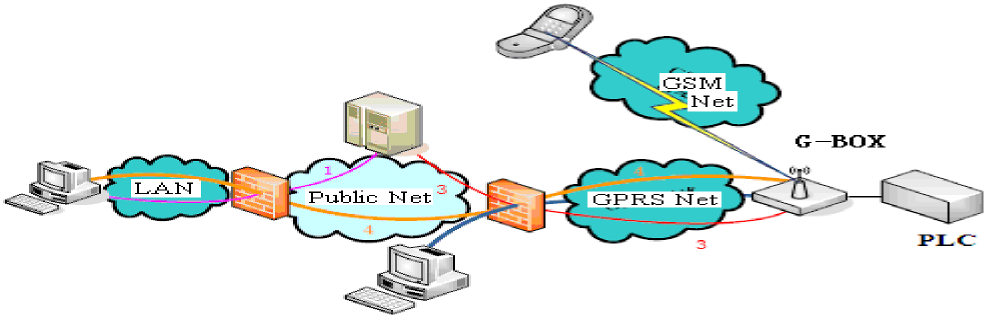


Figure 3.8: G-box communication

3.5.4 Applied field

- Industry control, remote sensing, remote measurement.
- Monitor pipe network.
- Collect and monitor oil field and coal mine data.
- Fault diagnoses of huge equipment.
- Any other situations need wireless data transmission.

3.5.5 The G-box setting

When setting the parameter, G-box receives data from COM port; after exiting parameter setting and return to working status the parameter become effective.

The parameters setting of the G-box are given in Table 3.1.

Table 3.1: The G-box parameters setting

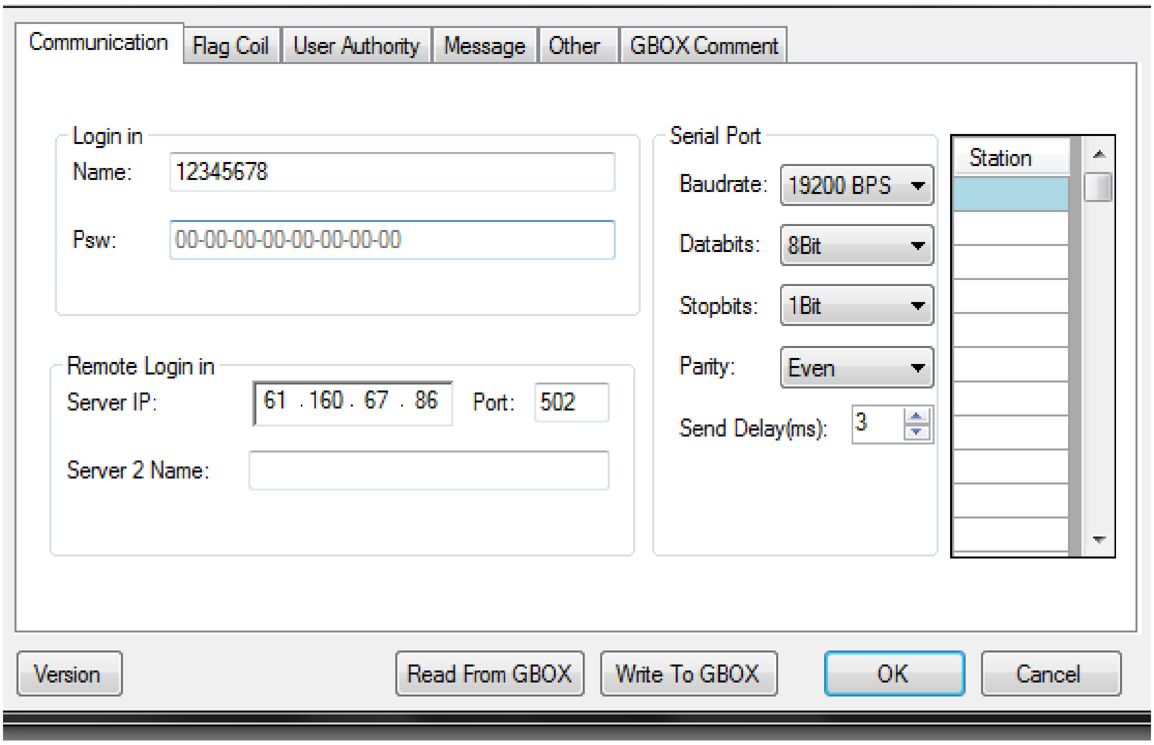
S1	S2	S3	S4	State	Description
Random	OFF	OFF	ON	Parameter setting	Use PC to set G-BOX original parameters
ON	OFF	OFF	OFF	Permanent online	G-BOX has already logged on the server
OFF	OFF	OFF	OFF	Dormancy	G-BOX is dormant, it will log on the server after phone call activation

When setting the parameter finished the LED indicator light function is shown in Table 3.2.

Table 3.2: LED indicator light

LED	Indicate	Function
PWR	Power	On available
COM	COM port	Glitter available
ONLINE	Connection	On available
GPRS	Mobile phone part works	Glitter once per second: searching for network Glitter once per three seconds: GSM connected Quickly glitter: GPRS data transmission On: start or abnormal

User's mobile number communicates with a G- box module to send and receive message with PLC through GSM network as shown in Figure 3.9.



Login in
Name: 12345678
Psw: 00-00-00-00-00-00-00-00

Remote Login in
Server IP: 61 . 160 . 67 . 86 Port: 502
Server 2 Name:

Serial Port
Baudrate: 19200 BPS
Databits: 8Bit
Stopbits: 1Bit
Parity: Even
Send Delay(ms): 3

Figure 3.9: G-box setting

CHAPTER FOUR

EXPERIMENTAL WORKS AND RESULTS

4.1 Introduction

The chemical system is composed from several major components, which are shown in Figure 4.1. This system consists of three tanks contains three types of acids (Nitric acid, Sulfuric acid, and Oleum), one big tank for mixer, pumps 24V DC, four sensors, solenoid valve PLC (Xinje), four 24V DC relays, and power supply 24V DC. Also PLC connected with wireless communication module (G-box) and HMI to operate the system from control room.

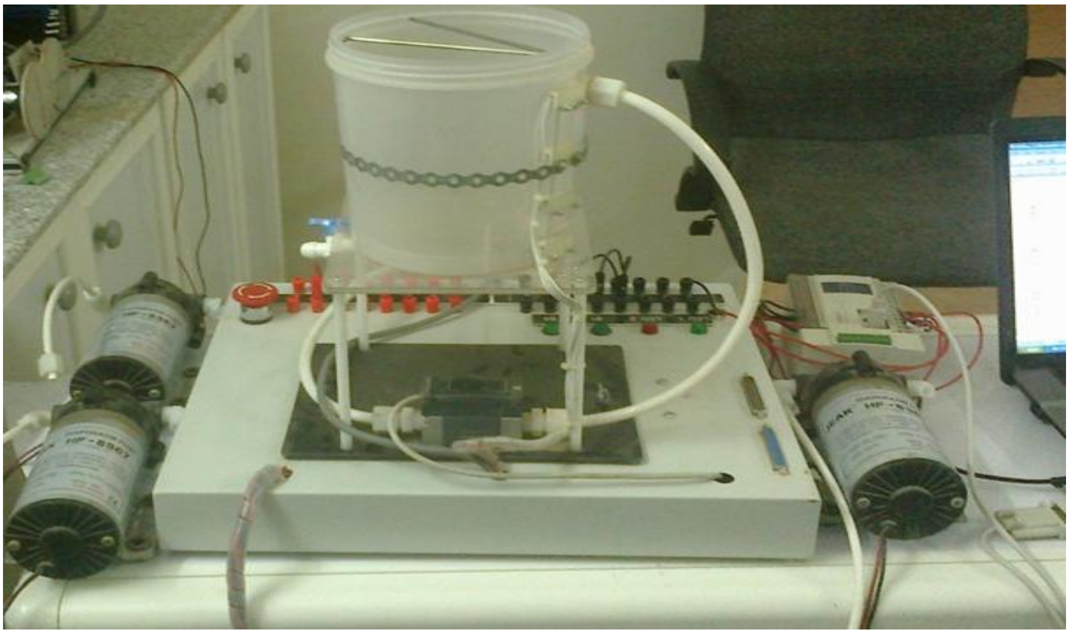


Figure 4.1: The overall hardware and software chemical system

4.2 Sequence of Operation

This system focuses on changing the classic technique in handling acids to a modern technique to maintain safety operation, accurate concentration for these acids and save of time. For this, all operations must be done automatically by just press start auto button from the screen as shown in Figure 4.2.

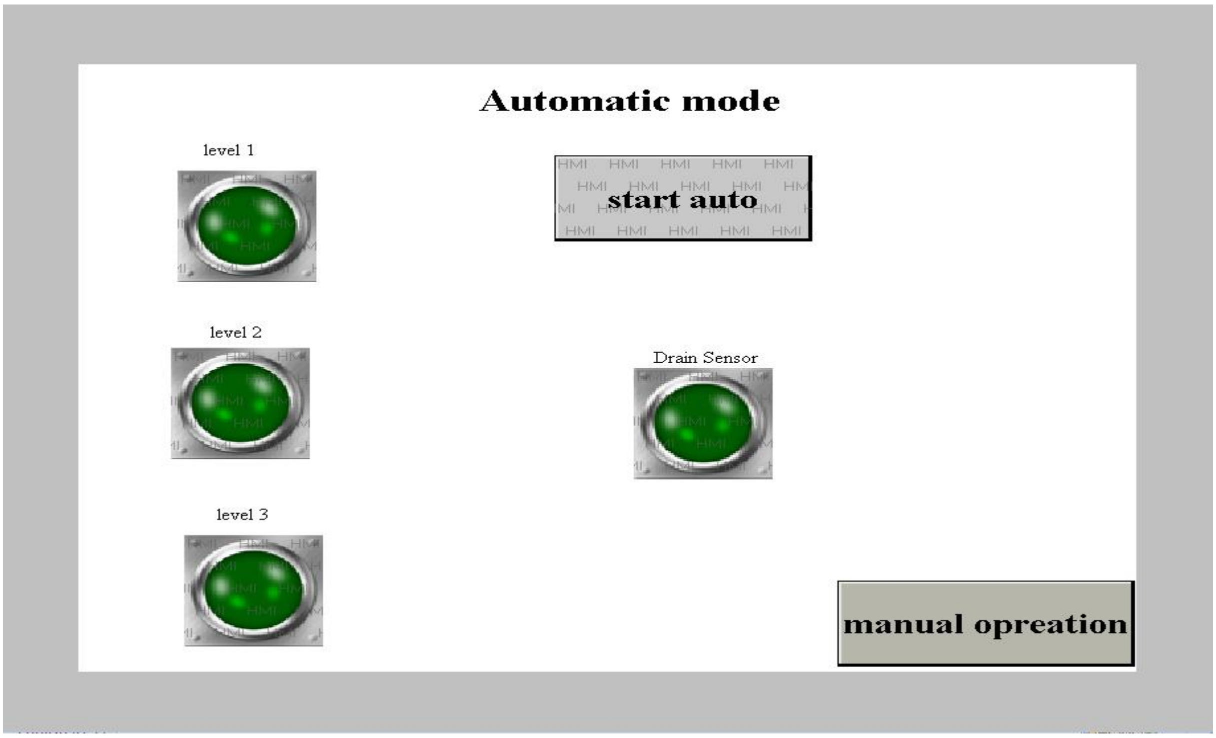


Figure 4.2: Automatic operation modes

And for maintenance used manual mode as shown in Figure 4.3.

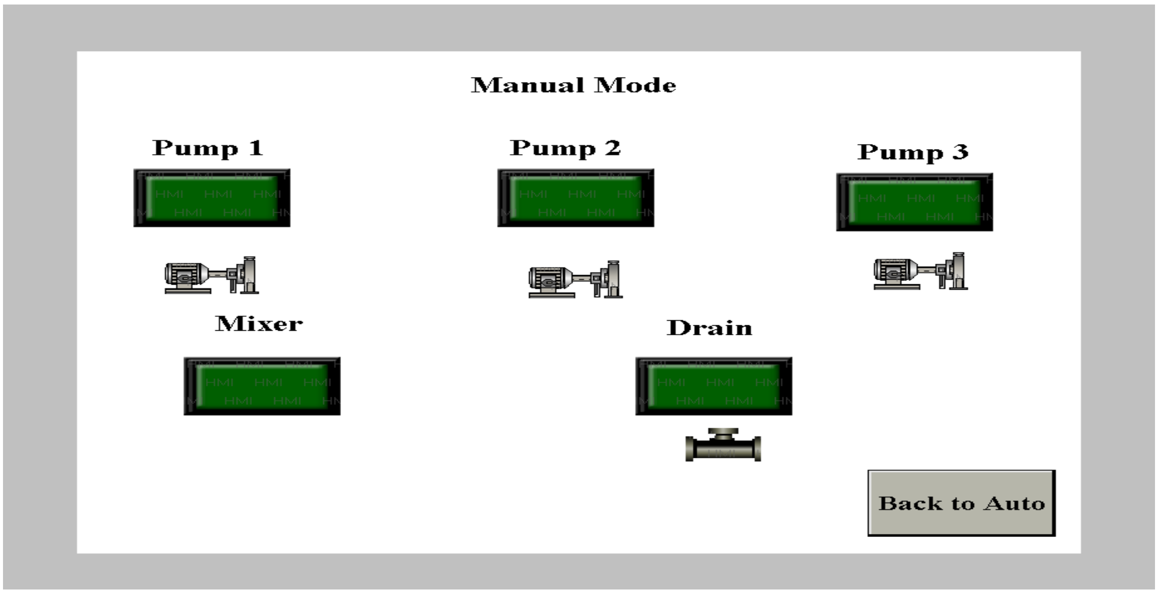


Figure 4.3: Manual operation modes

The steps used to mixt the three chemical acids are as follows:

Step 1: Pump 1 draw off nitric acid from tank 1 to the mixer and stop when level of nitric acid reaches sensor 1 and open pump 2 as shown in Figure 4.4.

Figure 4.4: Levels 1 nitric acid

Step 2: Pump 2 draw off sulfuric acid from tank 2 and added to nitric acid in mixer and stop when level of sulfuric acid reaches sensor 2 and open pump 3 as shown in Figure 4.5.

Figure 4.5: Level 2 nitric acid and sulfuric acid

Step 3: After that Pump 3 draw off oleum from tank 3 and added to nitric acid and sulfuric acid in mixer and stop when level of oleum reaches sensor 3 and start the stellar as shown in Figure 4.6.

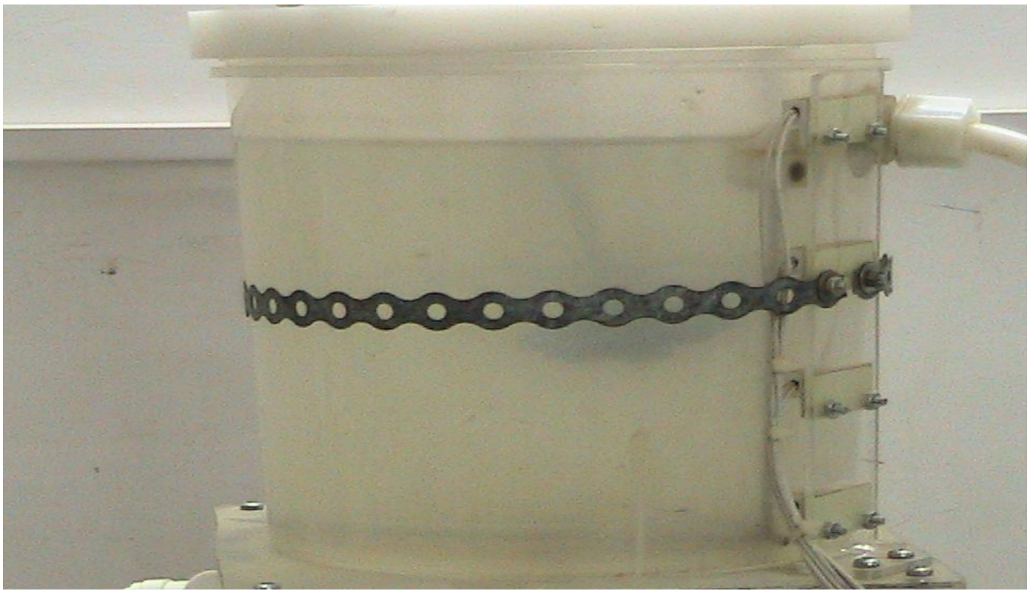


Figure 4.6: Level 3 nitric acid, sulfuric acid and oleum

Step4: When sensor 3 latch stellar in mixer start with timer until 20 second and mix these acids precisely as shown in Figure 4.7.

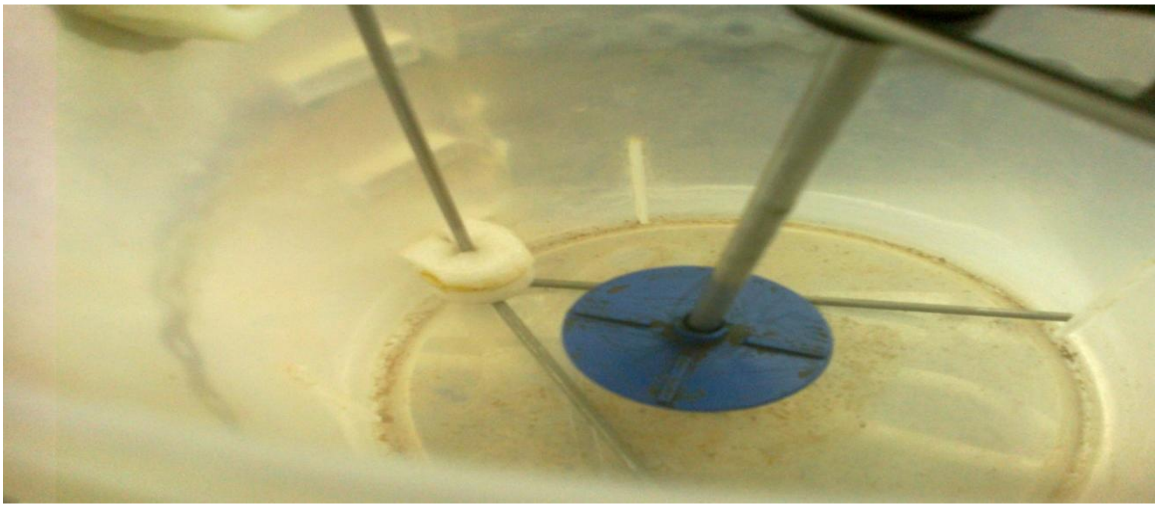


Figure 4.7: Mixture acids with stellar

Step5: After timer complete 20 second the solenoide valve open and drain the mixture acids to complete the chemical process in another phase.

4.3 The Flowchart

A flowchart is a pictorial representation that records, analyzes, and communicates information, as well as describes the operational process in a sequential manner, the Figure 4.8 shows the flowchart to indicate the steps of execute the program.

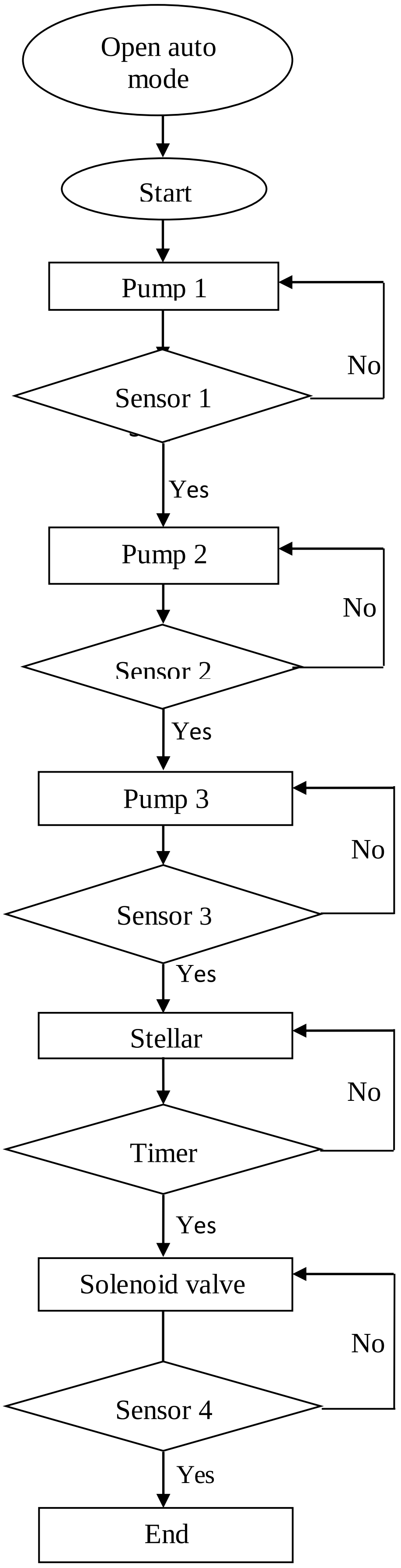
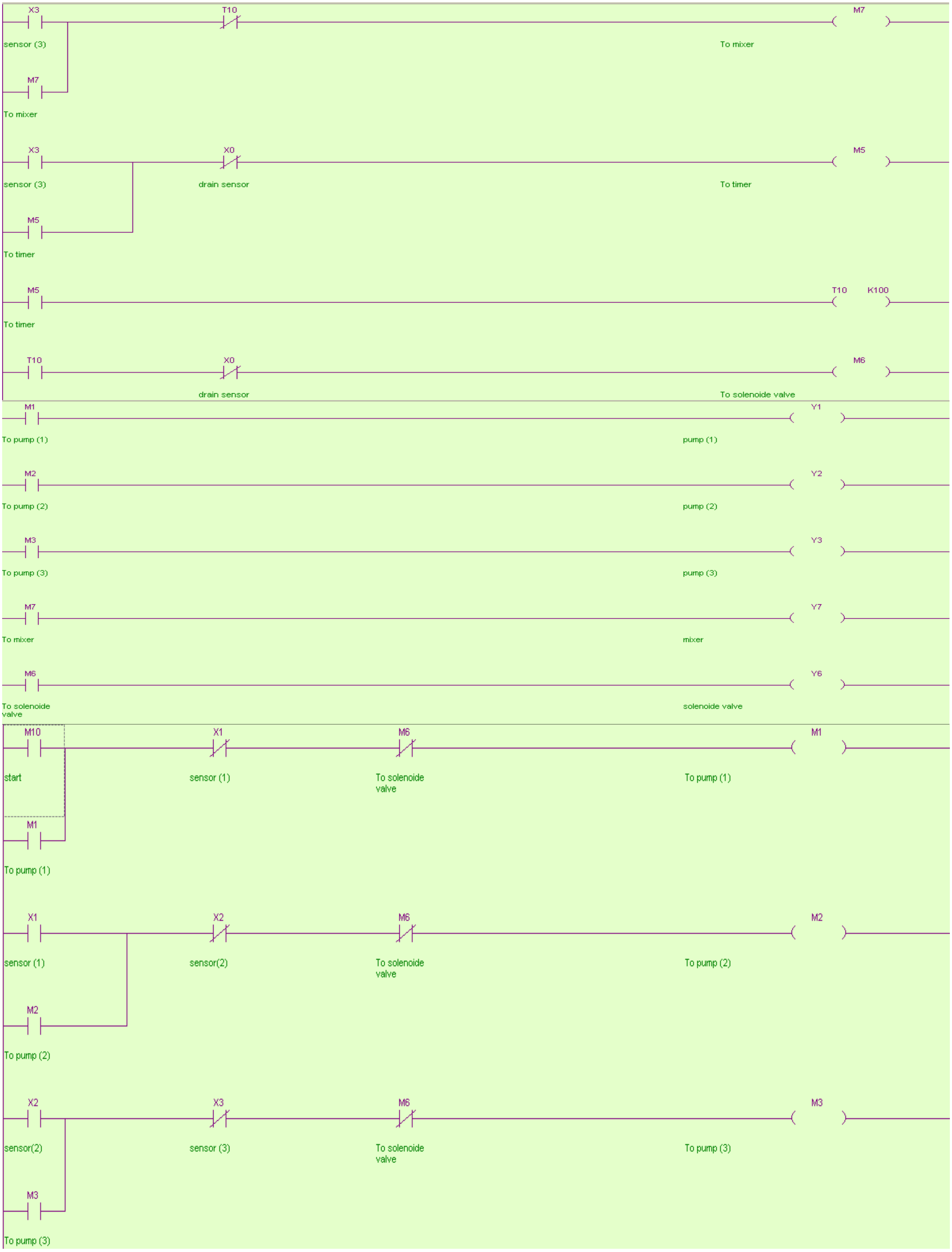


Figure 4.8: The flowchart

4.4 Ladder Program

The ladder program for the chemical system is given below.



CHAPTER FIVE

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The main goal of this thesis that to design an automatic chemical system to mixed three acids nitric acid, sulfuric acid, and oleum to produced nitric cellulose by change the classic manual method to modern method to maintain safety operation for operators like affect body contact, flammable, vapors... etc. Accurate concentration for these acids and Save of time for the operation.

This work has presented the use of the PLC via wireless communication via internet with touch screen to control the chemical operation remotely. The sensor is adjusted such that when the level of the acid reaches the sensor green lamp will appear on the screen HMI. The sensor change pump to another one. The communication between PLC and HMI do by wireless communication module (G-box) by internet.

5.2 Recommendations

For the development of this work, it is recommend to:

- Use a sensor with high accuracy. Or change the sensors to flow meters.
- Use mobile beside or instead of HMI to supervise the operation.
- Use SIM card with an excellent network.
- The speed of data transmission between the transmitter and receiver should be high.

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