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Microcontroller Based Rotary Evaporator for Solution Growth

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ABSTRACT: Crystal growth is an interdisciplinary subject. The main research work involved in this is the production of pure materials and improved equipment associated with the preparation of the materials. The main research work involved in this is the development of microcontroller based rotary evaporator for the solution growth. The PIC microcontroller PWM module is used to control the DC motor in turn it controls the operation of rotary evaporator equipment. This evaporator helps to mix up the reagents reaction and speed up the overall process. The rate of evaporation of the solvent is increased and there by seeding of the solution becomes better with the help of the DC motor. The PWM of the DC motor drive was adjusted at the different choice. The polarity of rotation is automatically selected by the toggle switch.

Keywords: DC motor; PIC Microcontroller; Solution growth; Nucleation; Solvent evaporation.

INTRODUCTION

Crystals are the unacknowledged pillars of modern technology. Crystal growth is an interdisciplinary subject. The main research work involved in this is the production of pure materials and improved equipment associated with the preparation of these materials. Evaporation is by far one of the easiest methods for crystallizing organic, inorganic and organometallic small molecule compounds. The choice of solvent is very important because it can greatly influence the mechanism of crystal growth and sometimes the solvent may be incorporated into crystalline lattice. The rate of crystal growth can be slowed either by reducing the rate of evaporation of the solvent or by cooling the solution. Organic reactions, however, tend to be long, slow, arduous processes that take hours, days, and sometimes even weeks to run to completion. To expedite the process, Crystallographers use solvents in their reactions. Solvents help to mix up the reagents of the reaction and make sure they come into contact more frequently, meaning that they will react more frequently, and thus speed up the overall process of the reaction.

There is a drawback to using solvents, however. When the reaction is finally completed, the solvent will still be mixed in with the product of reaction, meaning that the data concerning the product of reaction will be corrupted by the solvent. This isn't too much of a problem for a researcher, because it is possible when analyzing the product to discern which data relates to the compound made, and which data refers to the solvent that was used. In a professional pharmaceutical lab, however, one cannot present a drug which has made for testing if it still has solvent mixed into it. While solvents are great for moving chemical reactions along, they are not to be ingested and can often be harmful to the body. Therefore, it is necessary to find a way to completely remove the solvent from the product. One way of doing this is to use the rotary evaporator (rotovap).

MATERIAL AND METHODS

Background Study: Kaur and Chattergi[3] analyzed the efficiency loss of the dc motor. Steve Gorman[4] analyzed about the position of DC motor. C. C. Cheng[5] analyzed about the rotary evaporator solvent

efficiency. S. W. Chung and K. T. Chau[6] analyzed about the speed and torque variation of DC motor for PWM.

Problem Definition: Generally the rotational speed of a DC motor is proportional to the voltage applied to it and the torque is proportional to the current. The average voltage applied to the motor is varied by switching the supply voltage very rapidly. As the 'on' to 'off 'ratio is varied to alter the average applied voltage, the speed of the motor varies. But these can be done by manual control or ICs, we cannot control its speed in digital manner. To overcome this constraint, microcontroller is interfaced with DC motor. The PWM pulses generated from microcontroller is given to DC motor drive. By varying the pulse width on/off period of DC motor varies and which in turn varies the speed.

Objectives: This research work aims to develop a speed control of DC motor controlled by the PIC microcontroller which in turn used in the roto-evaporator to get the gentle evaporation of solvent from the sample. Thus increasing the rate of evaporation and best seeding of solution.

Nucleation: Nucleation is an important phenomenon in crystal growth and is the precursor of the overall crystallization process. Nucleation may occur spontaneously or it may be induced artificially. Nucleation is classified as homogeneous and heterogeneous nucleation respectively. Both these nucleation are called primary nucleation and occur in systems that do not contain crystalline matter. On the other hand, nuclei are often generated in the vicinity of crystals present in the supersaturated system. This phenomenon is referred to as secondary nucleation[1].

Methods of crystal growth: Growth of crystal ranges from a small inexpensive technique to a complex sophisticated expensive process. Single crystals may be produced by the transport of crystal constituents in the solid, liquid or vapour phase. On the basis of this, crystal growth may be classified into three categories as follows:

1. Solid Growth - Solid-to-Solid phase transformation
2. Liquid Growth - Liquid to Solid phase transformation
3. Vapour Growth - Vapour to Solid phase transformation

Evaporation: Evaporation is by far one of the easiest methods[2] for crystallizing organic and organometallic small molecule compounds. The choice of solvent is very important because it can greatly influence the mechanism of crystal growth and because the solvent may be incorporated into the crystalline lattice. It is customary to screen a large number of solvents or solvent mixtures to find the best conditions for crystal growth. The rate of crystal growth can be slowed either by reducing the rate of evaporation of the solvent or by cooling the solution. Formation of rosette-shaped masses is an indication of an insufficient number of nucleation sites. The number of nucleation sites may be increased either by seeding the solution or by scratching the exposed surfaces of the glass vessel.

Working of rotovap or rotary evaporator: The rotovap takes advantage of the low boiling points of solvents by creating an environment where the solvent will rapidly boil off leaving only the product. Conditions that are most conducive to boiling are: low air pressure and high temperature. The Rotovap works as such:

Round Bottomed Flask: This flask contains the product and the solvent that are trying to remove. It is attached to an axle that rotates the flask. This creates a thin layer of solvent mixture on the sides of the flask that will evaporate more quickly.

Heating Dish: The heating dish is filled with distilled water, which is heated and begins the evaporation process.

Condenser Coil: As the solvent begins to evaporate, the glass case surrounding the coil will begin to fill up with vapor. The condenser coil will help to condense the vapor back into liquid to avoid dangerous pressure buildups.

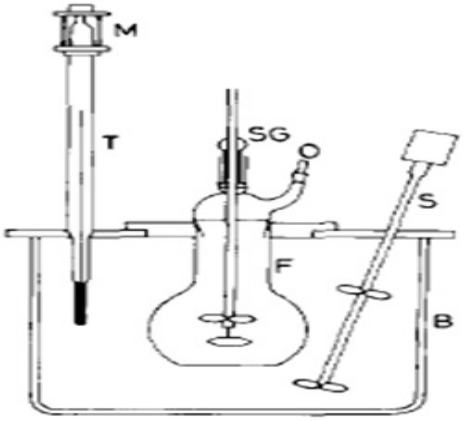
Water Hose: Attached to an external water source, the water hose runs cool water through the condenser coil and keeps it cool enough to allow the vapour to condense on it.

Collection Flask: When the evaporated solvent condenses on the condenser coil, it drips down into the collection flask.

Control Panel: This panel contains the controls for raising and lowering the round bottomed flask in and out of the warm water in the heating dish, the controls for the speed of rotation of the flask, and controls for how warm the water in the heating dish is.

The main advantage of rotary evaporation ensures gentle evaporation of solvent from the sample. Thus the rate of evaporation of the solvent is increased and thereby seeding of the solution becomes better. An opening is also provided in the setup to add any dopant while solution is kept for nucleation in the water bath.

The Figure 1 shows the diagram of typical rotary evaporator. The roto-evaporator apparatus used for solution growth technique to achieve proper nucleation during growth process the solution is poured inside the flask(F), which is then lowered into water bath(B). Solution is then stirred using stirring gland which can be rotated in both clockwise and anticlockwise direction.



Where: B – Bath; F – Flask; S – Stirrer; O – Opening; SG - Stirring Gland; T - Control Thermometer; M - Motor

Figure 1: Diagram of a typical rotary evaporator

To achieve proper nucleation during growth process the solution is poured inside the flask (F), which is then lowered into the water bath (B).Solution is then stirred using stirring gland which can be rotated in both clockwise and anticlockwise direction. The main advantage of rotary evaporation ensures gentle evaporation of solvent from the sample. Thus the rate of evaporation of the solvent is increased and thereby seeding of the solution becomes better. An opening (O) is also provided in the setup to add any dopant while the solution is kept for nucleation in the water bath

System Design: In this PIC 12F683 microcontroller is used as speed controller. Power supply unit, LED output unit, push button and toggle switch input unit, driver circuit for DC motor unit and DC motor interface unit are developed. This module shown in figure 2is connected with the control panel of the rotary evaporator. The software is written to interface DC motor, LED, Toggle switch. The toggle switch is used to select the polarity and PWM unit is used to control the speed of the DC motor.

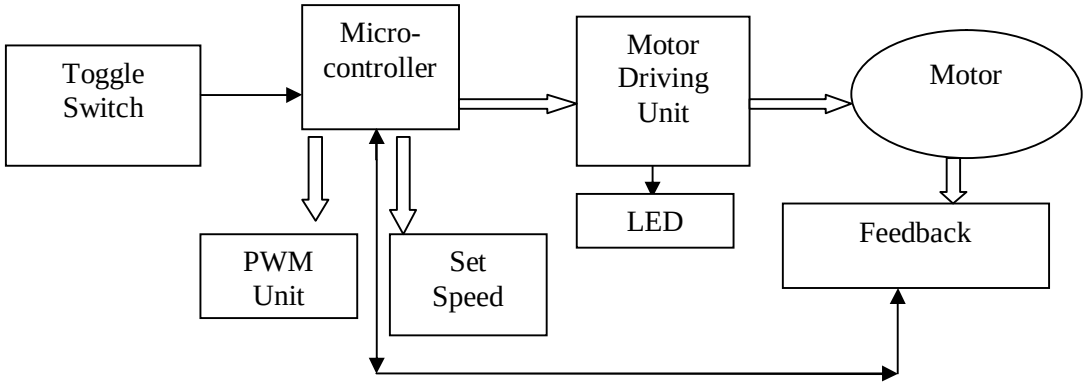


Figure 2: Hardware unit to control rotary evaporator

Experimental setup: Power supply is used to deliver DC power to the various units of microcontroller. The potentiometer is used for adjusting the speed of DC motor. Polarity of the DC motor can be changed by the toggle switch so that it changes the direction. Microcontroller PWM unit is used to generate the pulse width modulation pulses at the specified frequency range. The figure 3 shows the microcontroller based rotary evaporator for solution growth.

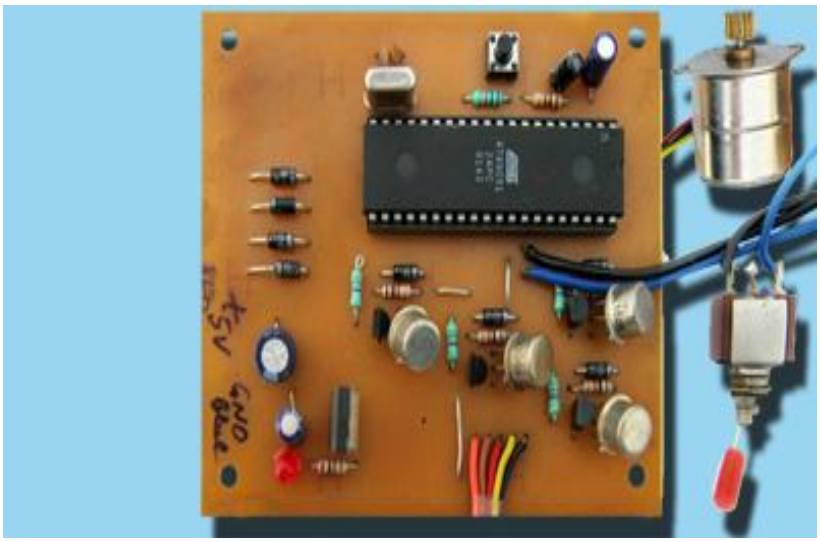


Figure 3: Micro Controller based Dc motor for rotary evaporator

RESULTS AND DISCUSSION

In the present study, Microcontroller based rotary evaporation for solution growth is compared with the manual roto-evaporator and IC based rotary evaporator. In the manual rotary evaporator, rate of solvent removed from the homogeneous solution is less than excepted result. The manual control is difficult in the absence of a lab assistant. In the IC based rotary evaporator, the rate of evaporation of the solvent is little bit increased with the manual control rotary evaporator. But, it is partially manual control. The on /off time of the DC motor and the polarity of rotation are not fully automatic.

The PWM unit in the microcontroller will control the on/off time of DC motor in the rotary evaporator for the solution growth if it is microcontroller based rotary evaporator. It is tested with the rate of evaporation of solvent and the seeding of the solution. The three different dishes are taken for testing. They are Acetone, water and Diethyl ether. The weights of the dishes are measured and the weights of dishes and substance also are measured before evaporation. After the evaporation the weights of dishes and substances are measured. The rate of evaporations is 100 percent in case of microcontroller based rotary evaporator for solution growth. The seeding is increased. The rotation of DC motor is automatically selected by the microcontroller and the on / off time with different choice could be done by the microcontroller. The facilities could be included in the microcontroller based rotary evaporator by modifying the software. It is fully automatic and needs no manual assistance.

CONCLUSION

The rate of evaporation of the solvent is increased and there by seeding of the solution becomes better with the help of the DC motor. The PWM of the DC motor drive was adjusted at the different choice. The polarity of rotation is automatically selected by the toggle switch.

The motor is operated by various PWM frequencies. The microcontroller based rotary evaporator for solution growth improves the function of the rotary evaporator. This evaporator helps to mix up the reagents and speed up the overall process. The rate of evaporation of the solvent is increased and there by seeding of the solution becomes better with the help of the DC motor.

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