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Design of Smart Hotstage-Microscopy for Enhancing Thermal Behaviour in Drug Melting Process

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Abstract – *The melting point of medicine is an important factor to determine the purity of medicinal raw materials, thermal behavior is closely related to the melting process of that. This research focuses on developing an intelligent Smart Hotstage-Microscopy prototype to observe thermal behavior in the drug melting process. This prototype heats the drug evenly on a hot plate equipped with a polarizing microscope, thermocouple sensor, microcontroller, and LCD display to view the temperature reading results. The performance of the Smart Hotstage-Microscopy prototype was obtained by testing the thermocouple sensor, heating system, cooling system, and drug validation. In this paper, the test results show that the thermocouple sensor test has an R^2 value of 0.9983, which shows that the correlation between the actual temperature and the hot part temperature shows a significant correlation, which is 99.83%. Testing the heating system from an average temperature of 28.57°C to the average temperature of 298.59°C took an average time of 11.28 minutes. Meanwhile, testing the cooling system took 5.19 minutes to reduce the temperature from an average of 298.59°C to 33.379°C. This test proves that the prototype of Smart Hotstage-Microscopy can work well in melting drugs.*

Keywords – *Drug Melting Point; Thermal Behaviour; Thermocouple Sensor; Smart Hotstage-Microscopy; Cooling System.*

I. Introduction

THE pharmaceutical industry plays an essential role in improving public health by providing effective and safe medicines to prevent, treat, or manage various diseases and medical conditions. Drug development and the complexity of drug formulations require an in-depth understanding of the thermal properties [1, 2]. Processing drug raw materials is a critical stage that requires an in-depth understanding of their thermal characteristics. Temperature is the main parameter that plays a central role in drug melting because it has a significant impact on the physical and chemical properties of drug raw materials [3].

Proper temperature regulation is the key to ensuring the smooth process of melting drug raw materials [4, 5].

Drug manufacturing in the pharmaceutical industry is closely related to the thermal behavior of materials. Therefore, thermal analysis becomes urgent to understand the characteristics of materials and to determine the melting point of drugs [6]. Drug raw materials with specific crystal shapes and specific melting points are essential in the process of making drug preparations [7]. The melting point is the temperature at which a solid substance turns into a liquid when heated. The melting point is an important characteristic in identifying the purity of drug raw materials. Melting of drug raw materials relies heavily on accurate temperature determination [8, 9]. If the temperature is too low, the drug raw material may not melt completely, causing inhomogeneity in the formulation and affecting the physical properties of the final product [10]. Meanwhile, temperatures that are too high have the potential to pose a risk of decomposition or undesirable chemical changes to the drug raw material [11, 12]. Therefore, monitoring the temperature at the melting stage is essential in ensuring that the process runs optimally and produces consistent drug raw materials [13]. Furthermore, the phase transformation from liquid into solid during the manufacture of medicinal raw materials is another important factor [14]. This process not only affects the physical structure of the material but also

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has the potential to affect the purity and stability of the drug. If the melted drug is not carefully monitored during the cooling process, there is a risk of crystallization which can harm the physical and chemical properties of the drug.

Although various thermal analysis methods are available, not many offer a smart innovation approach. Currently, drug melting point testing still uses the capillary tube method [15, 16]. However, this method has several weaknesses, including the compound undergoing sublimation before reaching the melting point, difficulty in determining the exact melting point, and rapid heating causing errors in determining the melting point. Another melting point testing tool uses Differential Scanning Calorimetry (DSC) [17, 18]. The weaknesses of DSC include its high cost, and the heating and cooling process of the DSC pad is carried out at a slow rate. Another test tool is the Hotstage, which is sold at an expensive price and requires liquid nitrogen cooling [19]. A Smart Hotstage-Microscopy tool is very useful in carrying out the initial stages of research on the characteristics of a drug or drug phase before continuing using an X-ray Diffraction (XRD) tool, which also requires relatively high costs. In addition, the advantages of smart sensor technology in the pharmaceutical industry and healthcare sector can improve control, accuracy, and efficiency in production processes and patient monitoring [20, 21]. This has a positive impact on product quality and overall healthcare.

Therefore, the Smart Hotstage-Microscopy prototype in this research focuses on improving the drug melting point observation device, which is carried out by heating the drug evenly over a hot cross-section. The device used to make these observations is known as the Smart Hotstage-Microscopy, which has a cross-section equipped with an internal heating element to provide heat evenly. Above the cross-section is an integrated polarizing microscope used to observe the melting and crystallization process of the drug. Apart from this, the Smart Hotstage-Microscopy prototype in this research is equipped with a thermocouple sensor to observe the heat conditions on the Smart Hotstage-Microscopy cross-section elements, and an ATMega 128A microcontroller is used as a heating control system, temperature reading, and cooling on the drug cross-section elements.

II. Research Methods

i. Drug Sample

Smart Hotstage-Microscopy is designed for accurate and efficient temperature regulation in heating and cooling processes. The performance of the smart hostage prototype in this research was applied to validate three drugs, namely gallic acid, paracetamol (PCT), and ferulic acid. The validation process of these drugs is critical to understanding the thermal response and temperature changes that may occur in the context of their use. The Material Safety Data Sheet (MSDS) is a guide in determining the melting point of drugs. Therefore, the Smart Hotstage-Microscopy heat sectional device that has been made can respond to the melting point value according to the MSDS.

Based on the MSDS, Gallic acid has a melting point of 251°C, paracetamol has a melting point of 168-171°C, and ferulic acid has a melting point of 168-172°C. Experiments were carried out by recording temperature changes in the hot cross-section, and the results were compared with the expected thermal parameters based on the MSDS. Melting point detection data on the Smart Hotstage-Microscopy matches expectations from MSDS information and provides strong validation of the thermal response of the Smart Hotstage-Microscopy system.

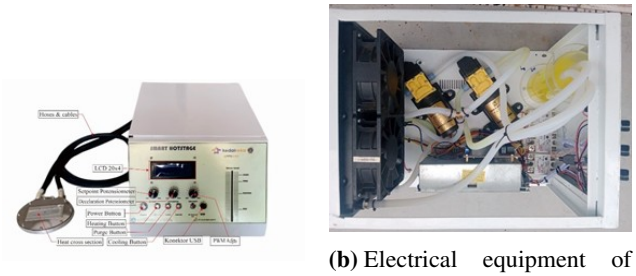
ii. Prototype Building

The device components for designing and building the Smart Hotstage-Microscopy Prototype consist of ATMEGA 128, aluminum, SSR, pump, heater, thermocouple sensor, resistor, power supply, LCD, push button, Arduino IDE, and toolkit. The prototype of a smart Hotstage-Microscopy was designed by detailing the functions and interconnections between components. This design includes selecting materials appropriate to the task of each component. All components are integrated to form a complete Smart Hotstage-Microscopy prototype. This process includes installing ATMEGA 128A as a system controller, SSR as an electronic switch, water pump, heater, thermocouple sensor, water flow path (hose), resistor as an electric current resistance, voltage source (power supply), LCD to display information, cables as component interconnections, and push buttons to control power on and off.

A laptop and Arduino IDE are used as Arduino programming software to support design creation. The Smart Hotstage-Microscopy prototype design was developed by utilizing the programming capabilities of the ATMEGA 128A microcontroller. This process ensures that each component is integrated efficiently and can be managed via the control system. Next, assemble the components using an electric soldering iron and toolkit. Each component is installed according to the design, with special attention to secure connections and fastening. This assembly process is a critical step in forming a hostage prototype that is functional and

well-connected.

A series of tests were conducted to evaluate the performance of the Smart Hotstage-Microscopy prototype. Testing includes verification of temperature control capabilities, electronic switch operation, water flow, and heating according to specified parameters. Testing also includes checking the performance of the temperature sensor, LCD, and the On-Off signal function of the push button. The results of the tests were analyzed to evaluate the performance of the Smart Hotstage-Microscopy prototype. The Smart Hotstage-Microscopy prototype can be seen in Figure 1.



(a) Smart Hotstage-Microscopy (outside view)

(b) Electrical equipment of Smart Hotstage-Microscopy (inside part)

Figure 1: Smart Hotstage-Microscopy prototype

iii. Hardware Design

The hardware system in this research is divided into three main parts, namely input, process, and output, which work together to ensure the heating and cooling processes in the Smart Hotstage-Microscopy heat section work well. The inputting of the process consists of a sensor on the cartridge heater as a heating element, a potentiometer, and a push button. In the process part, an ATMEGA 128A microcontroller is used. The output section is an LCD and an SSR.

The central component of this system is a highly sensitive thermocouple sensor to measure temperature with precision in the hot cross-section of the Hotstage. The setpoint potentiometer is the master in setting the temperature target value to ensure that the desired temperature can be achieved and maintained precisely. Furthermore, the deceleration potentiometer becomes a primary element to control deceleration in the temperature control process. By setting this value, the system can perform smooth temperature adjustments, prevent unwanted fluctuations, and maintain operational stability.

On the other hand, the PWM Adjust potentiometer has a crucial role in determining the rate of deceleration of the acceleration of temperature increase. With this setting, the system can respond quickly to desired temperature changes, providing comfort and efficiency in heat control. The role of the Atmega 128A as a microcontroller is as the smart brain behind the operation of the entire system. Carrying out the device controller and data storage role, the Atmega 128A ensures perfect cohesion and coordination between all the elements involved. The LCD (Liquid Crystal Display) is the main information window, visually monitoring temperature and ongoing processes. Users can easily combine and analyze temperature data in real-time.

Electrical load control is carried out by a Solid State Relay (SSR), which efficiently manages the flow of electricity to maintain system stability and security. Meanwhile, the Heating, Purge, and Cooling push buttons provide easy-to-use manual controls to activate crucial functions in the heating and cooling process. This system can create an optimal environment for heat addition and loss processes in the cross-section and offers maximum ease of use and control through the integration of scalable and advanced technology.

The electronic circuit based on hardware design can be seen in Figure 2. Wiring design is crucial to ensure device operations comply with the required technical standards. The Smart Hotstage-Microscopy system works on 12V DC and 220V AC power sources. The 12V DC voltage input performs various critical functions in the smart hotstage. Meanwhile, the 220V AC voltage, as an Alternating Current, provides essential support for the entire system's operation. The use of IO Pins connects and controls various components and devices within the hotstage, including temperature sensors, heaters, and cooling systems, through proper configuration.

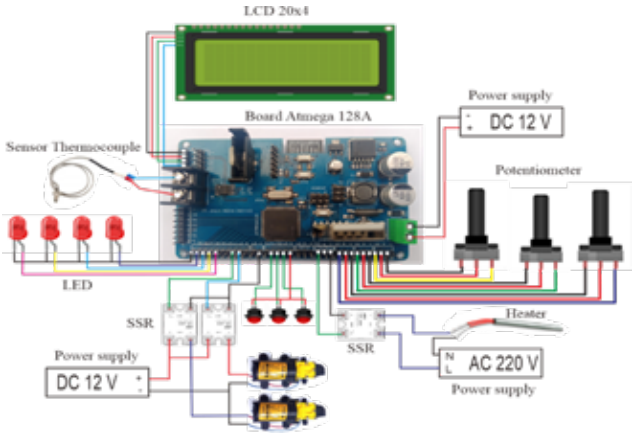


Figure 2: Electronic circuit design

iv. Software Design

The software design of the Smart Hotstage-Microscopy prototype consists of the main program that controls the heating and cooling process. The program is embedded in the ATMEGA 128A microcontroller and is responsible for managing temperature readings, system

responses, and user interactions. The software is designed to process input from the thermocouple sensor, execute control logic, and generate appropriate output signals to regulate the heating system.

The control algorithm follows a feedback mechanism where the thermocouple sensor continuously measures temperature and sends data to the ATMEGA 128A. The microcontroller then adjusts the heating system based on predefined setpoints. The software uses PWM (Pulse Width Modulation) signals to control the heater's intensity. A PID (Proportional-Integral-Derivative) controller is implemented to enhance precision and stability in temperature regulation.

User interaction is facilitated through an LCD display that shows real-time temperature readings. Push buttons are integrated to allow users to start or stop the heating and cooling process manually. The software logic is designed to ensure safety by incorporating emergency shutdown features when the temperature exceeds a predefined limit.

Figure 3 illustrates the software flowchart, outlining the system's operation, from initialization to monitoring and control.

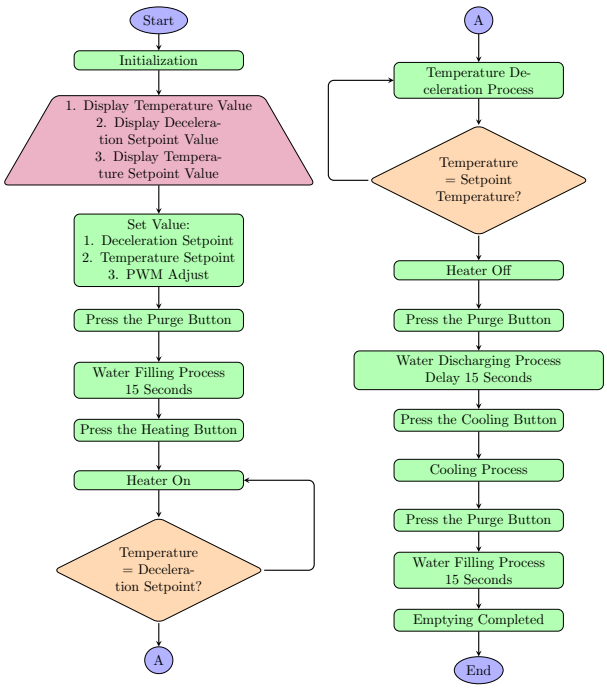


Figure 3: Software flowchart of Smart Hotstage-Microscopy

III. Results and Discussion

i. Performance Prototype Hotstage

The Smart Hotstage-Microscopy Prototype is a tool designed to analyze the thermal behavior of a drug during the melting process. This allows for determining the melting point of the drug, which can be read via a desktop computer or PC through a USB connection. The performance of the Smart Hotstage-Microscopy hot section prototype was evaluated through a series of trials, including testing the thermocouple sensor, heating system, and cooling system. The performance results are described in this section.

ii. Thermocouple Sensor Testing

During the testing phase, the thermocouple sensor was calibrated by comparing its readings from the hot cross-section with a pre-calibrated temperature sensor placed on a prepared glass surface. The purpose of calibration is to ensure that the thermocouple sensor provides accurate temperature readings that align with actual temperature values. Calibration is conducted by adjusting the temperature on the hot section (actual temperature) and the temperature above the glass preparation to obtain thermocouple sensor readings with minimal deviation.

The calibration temperatures used in this study were $50°C$, $100°C$, $150°C$, $200°C$, $250°C$, and $300°C$. The calibration results for the thermocouple sensor are illustrated in Figure 4.

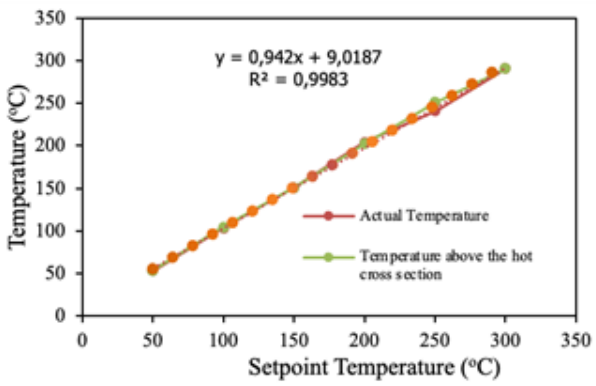


Figure 4: Calibration of thermocouple sensors

Based on Figure 4, the temperature difference between the actual temperature and the temperature on the hot cross-section is relatively small. The correlation between actual temperature and hot cross-sectional temperature was analyzed using the coefficient of determination (R^2), which quantifies the degree to which variability in the dependent variable is explained by the regression model, as given by Equation (1):

$$R^2 = 1 - \frac{SSE}{SST} \tag{1}$$

where:

- SSE represents the sum of squared errors (residual sum of squares).

- *SST* represents the total sum of squared variations in the dependent variable.

This regression model method has been previously applied by [22] to determine sensor accuracy in RH sensor calibration. According to [23], a calibration curve is a regression model used to predict the concentration of an unknown analyte based on an instrument's response to a known standard. The sensor calibration method using a regression model is a simple yet effective approach for sensor calibration [24]. Temperature sensor calibration using the regression method has also been validated by [24].

In this research, the coefficient of determination (R^2) measures how well variable y can be explained by variable x within the regression model. The R^2 value ranges from 0 to 1, where a higher R^2 value indicates a better regression model [25]. The results of the thermocouple sensor calibration show an R^2 value of 0.9983, indicating a significant correlation of approximately 99.83% between the actual temperature and the hot section temperature. Furthermore, this correlation analysis demonstrates that the thermocouple sensor exhibits high accuracy, making it suitable for measuring the melting point of drugs or other materials.

The obtained R^2 value is consistent with findings from [26], which evaluated various thermocouple sensor calibration methods and reported an average R^2 value of 0.999.

iii. Heating System Testing

The heating system in the Smart Hotstage-Microscopy is a critical parameter that must be identified to ensure proper functionality. Heating system testing is conducted to determine whether the Smart Hotstage-Microscopy undergoes heat changes. If no heat change occurs, the drug melting process will not be completed. Table 1 presents the results of heating system testing based on temperature changes over a certain period. A total of ten experiments were carried out to evaluate heat variations under different conditions.

Table 1: Test Results of the Heating System on the Hotstage

No	Trials	Initial Temp. (°C)	Final Temp. (°C)	Time (Minutes)
1	Trial 1	18	300	12:39
2	Trial 2	21	300	12:31
3	Trial 3	22.7	301	12:31
4	Trial 4	20	303	10:39
5	Trial 5	30	300	09:57
6	Trial 6	32	299	09:32
7	Trial 7	34	299.2	10:05
8	Trial 8	30	294.7	13:44
9	Trial 9	39	294	11:33
10	Trial 10	39	295	11:32
Average		**28.57**	**298.59**	**11:28**

Based on Table 1, the heating system test results demonstrate that the Smart Hotstage-Microscopy undergoes significant heat changes. The average initial temperature recorded was 28.57°C, with the highest temperature reaching 298.59°C. The average time required to achieve the temperature setpoint was approximately 11 minutes 28 seconds.

The testing of the heating system confirms that the initial and final temperatures in the Smart Hotstage-Microscopy can reach high values, which are relevant for drug melting processes or other thermal experiments requiring precise temperature control.

Trial 4 achieved a final temperature of 303°C relatively faster, in 10 minutes 39 seconds, whereas Trial 8 showed a slightly lower final temperature of 294.7°C with a longer heating time of 13 minutes 44 seconds. These variations may be attributed to differences in experimental parameters or initial conditions in each test.

The Smart Hotstage-Microscopy offers flexibility and control over heating, ensuring effective temperature regulation for specific applications. The ability to maintain consistent temperature changes and reasonable control over heating time is essential to ensuring experimental quality and reproducibility [27].

iv. Cooling System Testing

Testing of the cooling system in the Smart Hotstage-Microscopy system aims to determine the rate of heat reduction in the Smart Hotstage-Microscopy cross-section when the material melts. The maximum temperature used on the hot stage is 300°C, so the focus of testing the cooling system is on the time needed to reach the lowest temperature point. The cooling system test results can be seen in Table 2.

Table 2: Test Results of the Cooling System on the Hotstage

No	Trials	Initial Temp. (°C)	Final Temp.e (°C)	Time (Minutes)
1	Trial 1	300	30	05:53
2	Trial 2	300	31	05:57
3	Trial 3	301	31.5	05:32
4	Trial 4	303	39	05:26
5	Trial 5	300	32.7	05:20
6	Trial 6	299	34	05:45
7	Trial 7	299.2	34.7	05:24
8	Trial 8	294.7	32	04:06
9	Trial 9	294	35.5	04:25
10	Trial 10	295	33.39	05:27
Average		298.59	33.379	05:19

Based on Table 2, the test results show that the time required to reduce the temperature from a high of 300°C to a low of 30°C is around 5.53 minutes. Obtaining this data is significant in determining the speed of heat loss in the Smart Hotstage-Microscopy cross-section under specific conditions. The average cooling time for the trial was approximately 5:19 minutes.

Such an efficient heat loss rate is significant in various applications, especially in material processing

and laboratory trials. Reasonable heat loss control can improve operating efficiency and ensure consistent experimental results. These findings provide valuable insights for developing and improving thermal processes in Smart Hotstage-Microscopy sections for future applications.

Additionally, cooling system testing is influenced by the initial temperature. Experiments with higher initial temperatures tend to require longer cooling times. For example, Trial 4, with an initial temperature of 303°C, took 5:26 minutes, whereas Trial 8, with an initial temperature of 294.7°C, only took 4:06 minutes. This indicates a correlation between the initial temperature and cooling time, which should be considered when planning the heating and cooling processes.

IV. Conclusion

In this research, a Smart Hotstage-Microscopy prototype has been designed to analyze thermal behavior in the drug melting process. The results of Smart Hotstage-Microscopy readings can be accessed via a desktop computer or PC through a USB connection. Additionally, this prototype provides flexibility in heat control during the melting process. The overall performance of the Smart Hotstage-Microscopy prototype demonstrates good quality in measuring temperature and maintaining control over the heating and cooling processes. The thermocouple sensor calibration results showed a temperature difference between the actual temperature and the temperature above the hot cross-section, with a coefficient of determination (R^2) of 0.9983, indicating a significant correlation. The heating system test revealed that the time required to reach the average temperature setpoint was approximately 11 minutes and 28 seconds, demonstrating good flexibility and control of the heating system. Meanwhile, testing of the cooling system showed a relatively short time of about 5.53 minutes to reduce the temperature from 300°C to 30°C. Smart Hotstage-Microscopy validation with pharmaceuticals confirmed that the system is reliable for drug melting point testing in pharmaceutical industrial applications.

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