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Gage R&R study for the validation of a contact angle measurement system prototype

Estudo Gage R&R para a validação de um protótipo de sistema de medição de ângulo de contato

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Resumen

En el proceso de desarrollo de un nanorecubrimiento o un recubrimiento hidrofóbico, se debe medir y verificar distintivos parámetros para asegurar su eficacia y funcionamiento adecuado. Este procedimiento adquiere una importancia significativa, ya que determinará el éxito o fracaso del producto en comparación con sus competidores. Entre los parámetros relevantes se encuentra el ángulo de contacto entre el fluido y la superficie, el cual proporcionará información acerca de la capacidad de repeler el líquido por parte de la fórmula desarrollada por la empresa, lo cual sirve para determinar su calidad o la necesidad de perfeccionamiento. Por eso, la presente investigación tiene como objetivo proponer una alternativa de solución que sea mediante un estudio Gage R&R para que luego sea estandarizada para generar mejoras económicas. Para ello, se diseñó una investigación mixta de carácter explicativo y correlacional, con enfoque cuantitativo. A pesar de la existencia de un amplio campo de investigación y oportunidades de aplicación en el estudio de los ángulos de contacto en la actualidad, se observa una carencia de herramientas o soluciones específicas para estudiar y medir este principio, lo cual debe ser atendido para abordar el estudio y la evaluación de dicho fenómeno.

Palabras clave: ángulo de contacto, estudio Gage R&R, alternativa, recubrimientos hidrofóbicos.

Abstract

When developing a Nanocoating or a Hydrophobic Coating, the measurement and verification of different parameters is required to ensure its operation and efficiency, because this will mark the success or failure of the product compared to its competitors, one of them is the contact angle between the fluid and the surface, which will let us know how much the formula developed by the company repels the liquid, defining whether it is good or needs to be improved. This research seeks to propose an alternative solution validated through a Gage R&R study that can later be standardized and represents an economic improvement. It is a mixed investigation of an explanatory and correlational nature, and with a quantitative approach.

At present there is still a wide field of research and application opportunities in the study of contact angles. However, there are few tools or solutions to study and measure this principle, leaving gaps when studying and evaluating it.

Keywords: contact angle, Gage R&R study, alternative, hydrophobic coatings.

Resumo

Ao desenvolver um Nanocoating ou um Coating Hidrofóbico, é necessária a medição e verificação de diversos parâmetros para garantir seu funcionamento e eficiência, pois isso marcará o sucesso ou o fracasso do produto em relação aos seus concorrentes, um deles é o ângulo de contato entre o fluido e a superfície, que nos informarão o quanto a fórmula desenvolvida pela empresa repele o líquido, definindo se está bom ou precisa ser melhorado. Esta pesquisa busca propor uma solução alternativa validada por meio de um estudo Gage R&R que posteriormente pode ser padronizada e representa uma melhoria econômica. É uma investigação mista de natureza explicativa e correlacional, e com abordagem quantitativa.

Atualmente ainda existe um amplo campo de pesquisa e oportunidades de aplicação no estudo dos ângulos de contato. No entanto, existem poucas ferramentas ou soluções para estudar e mensurar esse princípio, deixando lacunas na hora de estudá-lo e avaliá-lo.

Palavras-chave: ângulo de contato, estudo de medidor r&r, alternativa, revestimentos hidrofóbicos.

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Introduction

Much research has focused on obtaining superhydrophobic coatings, which have a high-water repellency capacity. This interest is based on the consequences that the contact of water with the surface of certain materials can have, as well as the various applications that have emerged for this type of coating. Superhydrophobic coatings are materials with the ability to repel water that are characterized by reaching contact angle values equal to or greater than 150°, between different parameters (Adak *et al.*, 2019; Bayer, 2020; Lin *et al.*, 2020; Sharma *et al.*, 2020). This combination of properties allows a drop of water to be deposited on the coated surface to maintain a spherical shape, since its stability depends on the degree of hydrophobicity to allow relatively fluid sliding on the surface when tilted.

Research into this phenomenon began in the 1970s, and in the 1990s Wilhelm Barthlott patented the principle of the "lotus effect" using nanotechnology. The interest in emulating this natural effect was motivated by the search for surfaces that do not get wet and the ability to self-clean, although it is worth mentioning the need to further refine the characteristics of these coatings, including the hydrophobicity or contact angle (Conantec, 2018).

For this reason, the objective of this research is to propose an alternative solution, which will be validated through a Gage R&R study and subsequently standardized to represent an economic improvement. To this end, the correct definition of the system and its proper interpretation, supported by a complete understanding of the objectives and needs of the study, are crucial, since an inadequate writing of the system or a wrong interpretation can result in problems and affect the definition of the elements. which represent the inputs in the system. This, in turn, affects their participation and relationship in the process, and influences obtaining an output because of the transformation or use of the selected elements, along with the applied methodology.

Furthermore, the sampling or study may be carried out incorrectly, which would affect the quality of the information for subsequent analysis and presentation. In the specific case addressed in this research, the importance of defining, through a system methodology, the necessary elements that will be related and allow the transformation of the information to be sampled is highlighted. This is crucial to carry out a Gage R&R study, since the absence of adequate means to measure and control specifications in a process can result in failures in the measurements of possible variables, which would compromise the use of available resources.

The measurement system, therefore, represents the eyes through which quality is observed, hence it is essential to evaluate the behavior of the processes. In this sense, measuring instruments must carry out reliable measurements that do not generate false alarms in relation to production quality. In other words, without a reliable measurement system, it is impossible to determine if good quality production is being achieved (Vinasco Isaza, sf).

However, carrying out this study is not limited only to having the necessary material resources, as it also implies having specific knowledge that facilitates the understanding of the interrelationship between the various variables or factors that define the study. In this context, repeatability and reproducibility are key concepts, and are defined as follows:

- Repeatability: Indicates the extent to which variability in the measurement system is caused by the measurement device itself.
- Reproducibility: Refers to the extent to which the variability in the measurement system is attributable to differences between the operators involved in the process (Minitab, 2021).

Once an understanding of the relationship between the factors involved and their presence in the study, as well as their responsibilities, has been achieved, it is possible to identify the sources of variation present in the prototype of the contact angle measurement system. This step is essential to carry out necessary corrections, achieve its validation and use it effectively in the measurement of contact angles in the development of coatings.

Specifically, this research seeks to develop a system for measuring contact angles of a liquid on a surface. To this end, we seek to obtain results in the Gage R&R measurement study with a variation percentage of less than 10% with the purpose of proposing an alternative for those who need to carry out contact angle studies in specific situations.

Now, to propose possible solutions, the following questions were asked:

1. What elements have a direct participation in the system development process?
2. What factors are considered in the Gage R&R study?
3. How are the variables related to the factors considered in the study and how do they contribute to its validation?

Aim

Develop a system for measuring contact angles between a liquid and a solid surface, with results in the Gage R&R measurement study of less than 10% variation.

Methodology

Initially, a mixed research approach was chosen since it addresses both theoretical and practical problems. Likewise, documentary, and experimental research was used to carry out an initial review of validated information that served as theoretical support. Subsequently, based on the knowledge acquired, the necessary changes were defined and implemented through experimentation.

On the other hand, the explanatory research approach was chosen because the objective is not limited to describing or approaching the problem, but rather seeks to identify its underlying causes to explain the reason for the object of study. In addition, correlational research was used since it sought to measure the degree of relationship between two or more concepts or variables. In this case, we investigated how repeatability and reproducibility worked, analyzing their interrelationship and their impact in the Gage R&R study.

Finally, as a fundamental pillar of the experiments, and especially about their design, a quantitative research approach was adopted, supported by the experimental designs implemented.

When making decisions about the sample size with which to work and given the lack of sufficient variables to perform an exact calculation, it was based on the recommendation provided by the web support of the statistical tool used, which in this case was Minitab. For the Gage R&R study, Minitab 2021 suggests the following:

> Use a cross-measurement R&R system to evaluate variation in the measurement system when all operators measure each part of the study. To conduct this study, you must have a balanced design with random factors. Select at least 10 parts that represent the expected range of process variation. For the study, three operators measure the 10 parts, three times per part, in random order (para. 1).

Based on the above, the decision was made to maintain the same configuration of the experiment, since it was considered that there were only 3 operators, as well as time restrictions.

In this context, various experimental designs were carried out using Minitab. The objective was to carry out sampling with different runs, apply Gage R&R measurement studies and evaluate the results to achieve the proposed objectives. As previously mentioned, 3 operators participated in the experimental design, each measuring 10 pieces on 3 occasions. With this design, the corresponding studies were carried out.

Applying a systems-based approach, the formulation of the problem and its resolution were proposed as follows:

SYSTEM

Tickets

- Materials (substrate and fluid)
    - Substrate: Glass
    - Fluid: Deionized water
- Laboratory components
    - dropper
    - pH and ppm meters
- Softwares
    - Default camera app in Windows

  - Microsoft PowerPoint
  - ImageJ
  - Minitab
- Measurement structure
  - acrylic base
  - Substrate supports
  - Camera
  - Dark box with its “platform”
  - Dark Box Regulator Brackets
- Material characteristics
  - water pH
  - ppm of water

Process

- Water characterization procedure
- Preparation of materials
- Design and manufacturing of measuring structure
- Functional test of structure, camera, and software
- fluid deposition
- Photo shooting
- Photo editing
- Contact angle measurement
- Worksheet design in Minitab
- Data processing
- Measurement system validation

Departures

- Process and system available for improvement and/or standardization

Theoretical framework

Measurement systems

The term *system* refers to the combination of two or more elements, subassemblies and parts that are necessary to carry out one or more functions. In the case of measurement systems, the main function is the objective and empirical assignment of a number to the properties or qualities of an object or event, so that these can be described. In other words, the measurement result must meet three key characteristics: be independent of the observer (objective), be based on experimentation (empirical), and establish a correspondence between numerical relationships and relationships between the described properties.

The objectives of the measure can range from the surveillance or monitoring of processes to the control of a process, as in the case of a warehouse. It can also respond to specific needs of experimental engineering (Tapia Esquivias *et al.*, 2019).

In the scope of measurement systems, everything that influences the measurement of a part must be considered, including measurement devices, measurement procedures, and operators (Minitab, 2021).

Measurement data quality

The quality of measurement data is defined by the statistical properties of multiple measurements obtained from a measurement system operating under stable conditions. Suppose, for example, that a measurement system, operating under stable conditions, is used to obtain several measurements of a specific characteristic. If all measurements are “close” to the master value for that characteristic, the data quality is considered “high.” Analogously, if some or all measurements are “very far” from the master value, the data quality is said to be “low.” The statistical properties most used to characterize data quality are the bias and variance of the measurement system.

Bias refers to the location of the data relative to a reference (master) value, while variance has to do with the dispersion of the data. Much of the variation in a set of measurements can be due to the interaction between the measurement system and its environment, making the data difficult to interpret and making the measurement system less desirable. If the interaction generates too much variation, the quality of the data may be so low that the data are useless. Managing a measurement system largely involves monitoring and controlling variation. Among other things, this involves focusing on understanding how the measurement system interacts with its environment to ensure the generation of data of acceptable quality (Tapia Esquivias *et al.*, 2019).

Analysis of the measurement system

Measurement system analysis is a method for determining the acceptability of a measurement system, which is why it is a critical component in any process aimed at improving quality. For a continuous response variable, measurement system analysis is used to determine the proportion of the total variation that comes from the measurement system itself. In the case of an attribute response variable, this analysis is used to evaluate the consistency and accuracy of the raters.

In this sense, it is essential to carry out the evaluation of the measurement system before resorting to tools such as control charts, capacity analysis or other analyses. This is done for the purpose of verifying that the measurement system is accurate and accurate, and that the data generated is reliable (Minitab, 2021).

Accuracy and precision

The International Vocabulary of Metrology (VIM) defines measurement accuracy as the proximity between a measured value and a true value of a measurand. Thus, a measurement is considered more accurate the smaller the measurement error. Although it is often stated that a measurement is more accurate when it has a smaller measurement uncertainty, this is not always the case, as mentioned above.

On the other hand, the VIM defines measurement precision as the proximity between the indications or measured values obtained in repeated measurements of the same object, or similar objects, under specific conditions. These conditions are mainly called *repeatability* or *reproducibility conditions.* In many cases, the term *precision* is specifically associated with repeatability, that is, it is linked to the dispersion of repeated measurements. This dispersion is commonly expressed through statistical measures such as the standard deviation, variance, or coefficient of variation under the specified conditions (Tapia Esquivias *et al.*, 2019).

Accuracy and precision are two fundamental characteristics of an acceptable measurement system.

Accuracy

Accuracy refers to how close a measurement system's measurements are to the true value.

Precision

Precision in the context of a measurement system refers to how close the measurements are to each other. Importantly, a measurement system can exhibit any combination of accuracy and precision. The accuracy of a measurement system consists of three main components: bias, linearity, and stability. On the other hand, the precision of a measurement system includes two key components: repeatability and reproducibility. These components can be exhaustively analyzed through various studies of the measurement system.

Data integrity is intrinsically linked to the integrity of the measurement system. If problems related to accuracy and precision are identified, it is crucial to improve the measurement system to ensure the reliability of the data obtained (Minitab, 2021).

Gage R&R Studio

If a company does not have the appropriate means to measure and control the required specifications, it is possible that it will incur errors in measurements related to variables such as quantity, diameter, weight, resistance, humidity, hardness, among others. These deficiencies can have a direct impact on customer satisfaction and their interests, as well as generate errors that will affect the company's profits.

The measurement system in a company represents the eyes through which quality is evaluated, hence the measurement instruments must make reliable measurements that do not give rise to false alarms in relation to the behavior of the processes. The lack of a reliable measurement system prevents determining whether production is carried out with good quality (Vinasco Isaza, sf).

One measurement system analysis (MSA) method that is valuable in evaluating measurement system precision and estimating repeatability and reproducibility is the Gage R&R study. It helps answer fundamental questions, such as whether the variability of the measurement system is small compared to the variability of the process, how much variability in the measurement system is attributed to differences between operators, and whether the measurement system can discriminate between different parties (Tapia Esquivias *et al.*, 2019).

A measurement system R&R study helps investigate the following:

- Repeatability: How much of the variability in the measurement system is caused by the measurement device.
- Reproducibility: How much of the variability in the measurement system is caused by differences between operators.
- If the variability of the measurement system is small compared to the variability of the process.

- If the measurement system is capable of distinguishing between different parts (Minitab, 2021).

Gauge studies provide valuable information on process performance and are essential tools in quality control as well as identifying sources of variation. Therefore, these studies constitute an essential tool to acquire knowledge about the performance of the processes. The central objective of a Gauge R&R study is to understand the sources of variability present in a measurement process, which represents the basis for various applications, including efficient process operation, variability compensation, process performance evaluation, quality control and the isolation of defective products, especially in manufacturing environments (Castañeda Hernández *et al.*, 2021).

Signal to noise ratio

The number of distinct categories is a metric used in R&R studies of the measurement system to evaluate the ability of the measurement system to detect differences in the measured characteristic. Thus, it can be understood as the number of groups within the process data that the measurement system can discern, which represents the number of different categories that it can distinguish.

For these purposes, a value of 5 or higher is recommended, since a value less than 2 indicates that the measurement system is not adequately monitoring the process (Tapia Esquivias *et al.*, 2019).

On the other hand, the signal-to-noise ratio measures how the response varies in relation to the nominal or target value under different noise conditions (Minitab, 2021).

Measurement system variation

Like any other process, a measurement system is subject to variation, which may be of common or special cause. To control variation in the measurement system, it is necessary to identify the sources of variation and then eliminate or reduce these various causes associated with the measurement process. The possible sources of variation include measurement instruments, standards, procedures, *software*, environmental components, among others (Tapia Esquivias *et al.*, 2019).

Measurement system variation is the variation that occurs when measuring something. Specifically, this variation is the sum of the variation caused by the repeatability and reproducibility of the measurement system. Any component of the measurement system, such as a measurement device, procedure, or software, can be a source of variation (Minitab, 2021).

Stability

Measurement stability refers to the change in bias over time, which represents the total variation of measurements of the same part made at different times. This variation over time is known as *gradual change.*

To monitor the stability of a measurement process, a control chart can be used, which involves measuring a main or control part with the same system over time. When measurements are taken, points that remain within the limits indicate that the process has not undergone significant changes, while points outside the limits suggest a change in the process.

Knowledge of the equipment and measurement conditions is essential to identify possible special causes when the system shows instability (M initab, 2021).

Repeatability

Repeatability refers to the variation in measurements obtained with a measuring instrument when it is used multiple times by an evaluator while measuring the same characteristic on the same part. This variation represents the intrinsic capacity of the equipment itself. Although commonly called *a team variation*, this can be misleading. Repeatability, in fact, is the common cause variation in successive tests under defined measurement conditions. Therefore, the most accurate term to describe repeatability is the variation within the system when the measurement conditions are established and defined with the fixed part, instrument, standard, method, operator, environment, and assumptions.

In addition to within-team variation, repeatability will also encompass any variation coming from any condition in the error model (Tapia Esquivias *et al.*, 2019). Repeatability is the variation caused by the measuring device and is manifested when the same operator measures the same part repeatedly, using the same measurement system and under identical conditions.

To estimate the repeatability of a measurement A, operator 1 performs 20 single-piece measurements with measurement system A. Similarly, to estimate the repeatability of a measurement B, operator 1 performs 20 single-piece measurements with measurement system B. By comparing the results, it can be determined that measurement system A exhibits less variation, indicating that it is more repeatable compared to measurement system B (Minitab, 2021).

Reproducibility

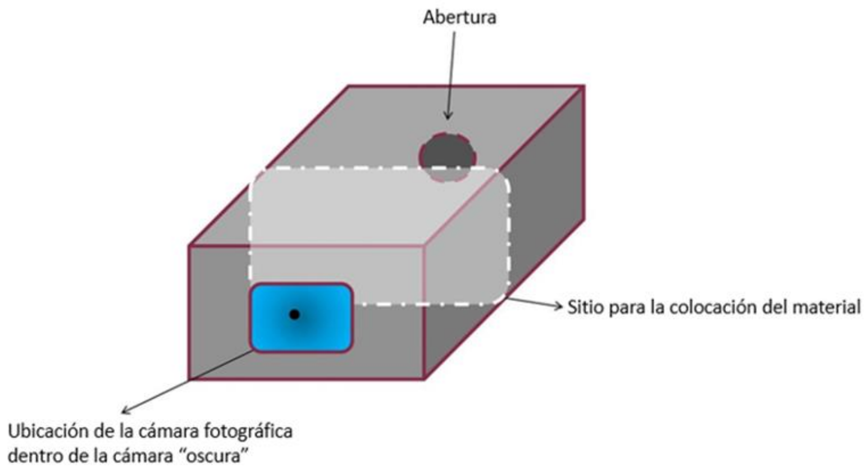
Reproducibility refers to the variation caused by the measurement system. This variation is evident when different operators make repeated measurements of the same part, using the same measurement system and under identical conditions.

In a scenario where operators 1, 2 and 3 measure the same part 20 times with the same measurement system, reproducibility can be evaluated (Minitab, 2021). Traditionally known as "inter-rater" variability, reproducibility is generally defined as the variation in the average of measurements made by different raters using the same measuring instrument when measuring the same characteristic on the same part. This concept is especially applicable to hand instruments influenced by the skill of the operator. However, in automated measurement processes, where the operator is not a significant source of variation, reproducibility is considered as the average variation between systems or between measurement conditions (Tapia Esquivias *et al.*, 2019).

Results

The development of a prototype (figure 1) for a contact angle measurement system was achieved, previously validated through a repeatability and reproducibility study. This prototype uses a specially designed "dark" camera to capture high-quality images. Supported by measurement *software,* this system offers a more economical and practical option when evaluating or measuring the contact angle during the development or use of a hydrophobic coating.

Figure 1. First design of the "dark" camera



Note: First design that was made for a better understanding and visualization of the "dark" camera that needed to be created, where the elements used are presented.

In relation to the statistical analysis used for validation, favorable results have been obtained that support the aforementioned. A percentage of variability that meets the required standards was demonstrated, achieving a figure of less than 10% variation, as evidenced in figures 2 and 3.

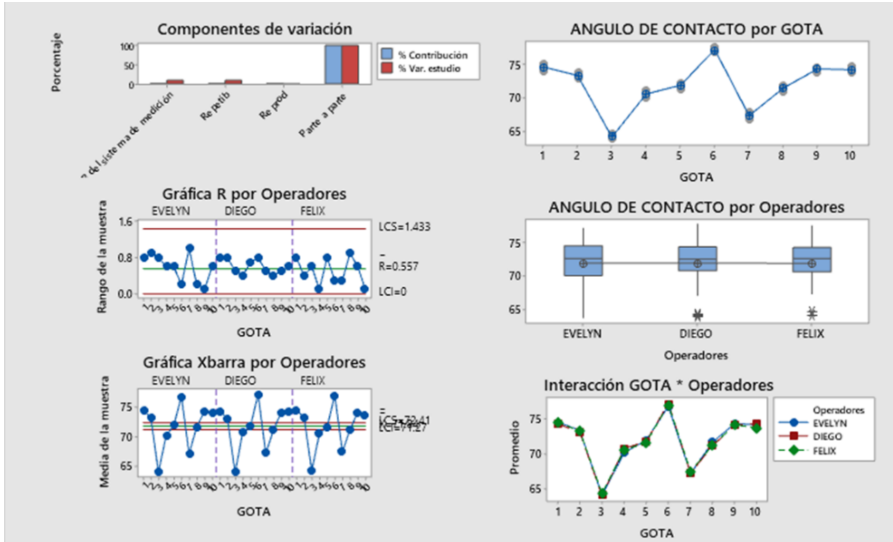
Figure 2. Evaluation of the measurement system

Evaluación del sistema de medición

Fuente	Desv.Est. (DE)	Var. estudio (6 × DE)	%Var. estudio (%VE)
Gage R&R total	0.34109	2.0466	8.99
Repetibilidad	0.34109	2.0466	8.99
Reproducibilidad	0.00000	0.0000	0.00
Operadores	0.00000	0.0000	0.00
Parte a parte	3.78026	22.6815	99.60
Variación total	3.79561	22.7737	100.00

Note: A variation percentage of 8.99 is seen in the study, which demonstrates that the structure and system developed is viable for use in measuring the parameter studied and analyzed.

Figure 3. Measurement System R&R Report



Note: It is possible to visually appreciate the results of the study in the different metrics obtained regarding the variation of the study. Software used: Minitab.

The above demonstrates favorable behavior with low variation and an excellent result in the "side-by-side" comparison, since this last parameter indicates that the system allows differentiation between the various samples analyzed. Furthermore, a behavior with low variation and a high correlation between the different operators and the technique used is observed.

Discussion

Initially, unfavorable results were obtained that required corrections and improvements, since they did not allow the validation of the structure and the measurement system. However, modifications were made to the system and contact angle measurement techniques to obtain better results and validate the structure and measurement system. This process allowed us to understand the different factors that influenced the variation, understand why corrections were necessary and improve the understanding of the operation and relationship between all the elements of the system.

After several corrections, it was possible to validate the measurement system along with its structure, as well as establish various standardized processes in a first version. A first training test was also carried out for measuring angles. Currently, the measurement system and structure can be used for future contact angle studies in a cost-effective and accessible manner at any location that meets the above specifications. Additionally, they remain open to further improvements and adaptations.

In the present study, the results obtained in relation to contact angles show notable similarities with previous studies, supporting and strengthening previous observations in the scientific literature. Some of the most notable similarities and associated implications are discussed below:

Coherence on Homogeneous and Heterogeneous Surfaces:

The results reveal significant consistency with previous research regarding the influence of surface character on the contact angle. Homogeneous surfaces exhibit behaviors consistent with theoretical expectations, while heterogeneous surfaces present notable variations, supporting the importance of surface composition in wettability.

Effect of Surface Energy:

The relationship between the surface energy of the surface and the contact angle aligns with previous findings, suggesting that interfacial properties play a key role in the interaction between the substrate and the liquid. This pattern highlights the relevance of considering surface energy as a significant predictor in materials design.

Influence of Surface Roughness:

Surface roughness emerges as a determining factor, corroborating previous results that indicate a direct relationship between roughness and the decrease in the contact angle. This phenomenon is attributed to the larger contact area and associated capillary effects, which has important implications in applications involving rough interfaces.

Validation of Theoretical Models:

Comparison of our results with existing theoretical models supports the validity of such models in predicting contact angles under various conditions. However, it is noted that certain deviations could indicate the need for specific adjustments in particular contexts.

Technological Applications:

The consistent findings offer significant opportunities for technological applications. A deeper understanding of contact angles can be leveraged in the design of water-repellent surfaces, efficient adhesives, and microfluidic devices, among others.

Taken together, the convergence of our results with previous research reinforces the robustness and applicability of the concepts related to contact angles. These results contribute to the existing body of knowledge and provide a solid foundation for future research and practical applications.

Conclusions

In conclusion, it can be indicated that there are opportunities for improvements in the training system for measuring angles in the *software*, especially in the foundation of the issue of pixels and their importance and usefulness when selecting points in the image.

Likewise, it is suggested to improve the dark box by incorporating an upper gate to facilitate the deposition of drops without having to remove the entire box, which would make the operation more practical and provide greater stability to the box. In addition, a study could be carried out to find a more resistant and durable material, adapted for everyday use in industrial or specific environments.

Another potential improvement would be the incorporation of a lens or magnifying glass that magnifies before taking the photo to avoid loss of image quality when zooming during editing, which would eliminate the need to crop the image.

Future lines of research

The contact angle, a measure of the interaction between a liquid droplet and a solid surface, is influenced by the physical and chemical properties of the liquid, solid, and intermediate medium. This parameter is crucial to analyze phenomena such as wetting, adhesion, capillarity, as well as mass and heat transfer. Although there are several methods for measuring contact angle, such as hanging drop method, skewed drop method, floating ring method, and optical method, these approaches face limitations. These include the need for specialized equipment, difficulties in obtaining clear images, sensitivity to external disturbances, and the complexity associated with data analysis.

Therefore, it is proposed to develop a contact angle measurement system that is simple, economical, accurate and robust. In this sense, some lines of research suggested for this project include the following:

- Design and build a portable device capable of generating droplets of different sizes and shapes on a solid surface, using materials such as syringes, needles, valves, or pumps.
- Develop an algorithm that can process digital images of droplets, extracting the contact angle value using techniques such as filtering, thresholding, contouring, segmentation, and regression.

- Apply the measurement system to various types of liquids and solid surfaces, analyzing the influence of factors such as temperature, pressure, chemical composition, and roughness.
- Investigate the possible applications of the measurement system in areas such as biomedical engineering, nanotechnology, textile industry and agriculture.

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