Novel Testing Method:
Checking the Efficacy of Protective Paint Coatings

(Design and Innovation, Electrolab India)

Annika Shah, Aditya Birla World Academy

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Overview:

Electrolab India, an established manufacturer of pharmaceutical testing equipment, encountered a significant challenge with corrosion in its dissolution testers, primarily caused by the highly acidic environment required for dissolution testing. This study aimed to find novel alternatives to address the limitations of traditional corrosion resistance tests (industrial standards), such as the ISO 9227 salt spray test, which failed to accurately simulate the conditions inside a dissolution tester due to the neutral pH of the $NaCl$ solution used. In overcoming this, a novel testing system was designed, developed, and tested with the objective of successfully evaluating the efficacy of protective coatings against corrosion. The system used hydrochloric acid (HCl) fumes, better mimicking acidic conditions dissolution testers are usually exposed to. The custom setup involved an innovative design, incorporating a closed system with rotating or oscillating capabilities to emulate fluid motion and account for hydrodynamics while ensuring the use of non-corrosive materials in the apparatus itself. Experimental verification showed that samples exposed to HCl exhibited higher corrosion rates than those in $NaCl$, confirming that HCl is a superior discriminatory agent. The results indicate that the new testing system is a more effective and unbiased tool for assessing the durability of coatings, offering Electrolab a reliable means to enhance its product lifespan and quality.

Background and Context – About Electrolab India:

Electrolab, established in 1984, manufactures pharmaceutical testing equipment and peristaltic pumps which used in several industries, exporting 50% of its production worldwide. The company has a well-trained production team supported by in-house CAD, software, hardware design engineers and well established vendors to support production target and a management that understands the importance of quality and timely delivery. Of the testing equipment manufacturers, dissolution testers account for 55% of total revenue.

Section A: Innovation and Design

The Dissolution Tester:

A tablet dissolution tester is a laboratory apparatus used to measure the rate at which an active pharmaceutical ingredient (API) is released from a solid dosage form (e.g., tablet) into a dissolution medium. Dissolution testing is crucial in the pharmaceutical industry to ensure that a tablet will release the drug at the right rate when ingested, which affects the drug's bioavailability and therapeutic efficacy.

The key components of a dissolution tester are:

1. Dissolution Vessel: Holds the dissolution medium (liquid), typically water or simulated gastric fluid.
2. Paddle or Basket: Stirs the medium to simulate the fluid motion in the gastrointestinal tract.
3. Thermostatic Water Bath: Maintains the temperature of the dissolution medium.

It is necessary that dissolution testers mimic the conditions of a human body. Thus, the water bath is always set to 37ºC (±0.5ºC), which is the average temperature of the human body. The pH of the dissolution medium depends on the part of the body where the drug is expected to dissolve. Most tablets dissolve in the stomach. Gastric juice (digestive fluid containing hydrochloric acid, digestive enzymes, mucus, and other substances) produced the stomach is of pH1.2 due to the presence of hydrochloric acid. Thus, it is important that the dissolution medium is kept at pH1.2 to mimic these highly acidic conditions.

Fig. 1 below is an image of a typical ‘Veritas-14’ dissolution tester manufactured by Electrolab.

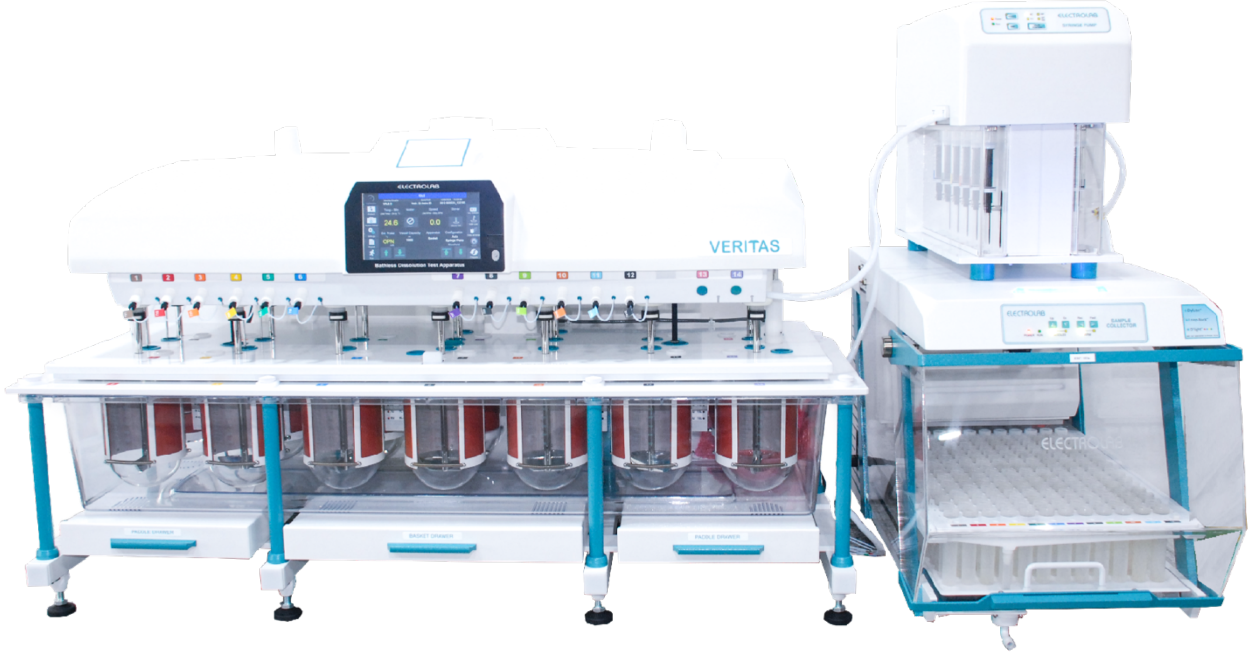

Fig. 1: Veritas-14 Dissolution Tester, ElectroLab

The Problem:

Hydrochloric acid (HCl) is classified as one of the strongest acids, having the ability to completely dissociate in water. It has a high corrosion rate of $\leq 0.1mm/a$ when the concentration of hydrochloric acid is $27\sim30\%$ and the temperature is $50\sim70^{\circ}C$. In the case of room temperature and any concentration of hydrochloric acid, the corrosion rate is $< 0.031mm/a$[1]. Due to this, Electrolab has found that many of their dissolution testers undergo corrosion over time. The highly acidic medium causes its HCl fumes to permeate the protective paint coating, in turn causing the mild steel to corrode. This decreases the durability of the dissolution tester and increases costs for Electrolab. More importantly, corroded equipment cannot be used in a sterile pharmaceutical laboratory environment. Fig. 2 and Fig. 3 show visible effects on the dissolution tester (DT).

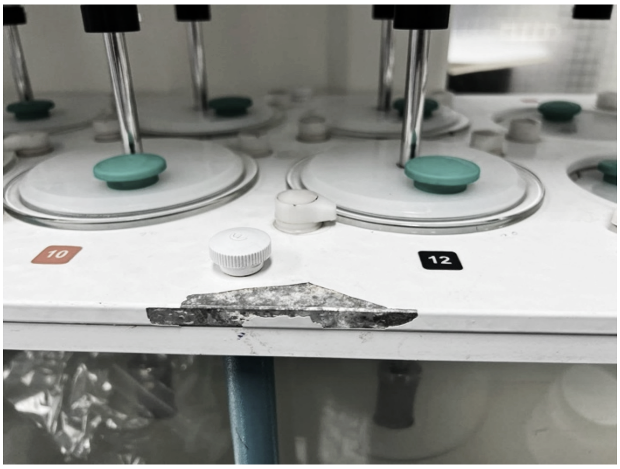

Fig. 2: Effect of HCl *on DT (1)*

Fig. 3: Effect of HCl *on DT (2)*

This problem was observed regularly in similar practical situations and Electrolab used to get regular complaints from its clients. Their first step was to improve the quality and composition of their protective paint coating. Initially, the firm used an epoxy-polyester coating with epoxy resins, polyester resins, and additives such as flow agents and UV stabilizers. On seeing poor results, a new coating was formulated, called the 'Multi Shield' polyester coating, with enhanced additives and composition. However, when *both* types of paint coatings were tested for their protective abilities, they consistently returned successful results. This revealed a flaw in the testing process, having already established one of the coating's practical ineffectiveness.

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[1] Fan, R., Zhang, W., Wang, Y., Chen, D., & Zhang, Y. (2021). Metal material resistant to hydrochloric acid corrosion. Journal of Physics Conference Series, 1732(1), 012134. https://doi.org/10.1088/1742-6596/1732/1/012134

The testing process followed was ISO 9227[2], the industrial standard 'salt spray test', which is a standardized test method used to evaluate the corrosion resistance of materials and surface coatings. It involves placing the coated samples in a chamber, where they are exposed to mist of 5% sodium chloride ($NaCl$) solution at $35{\sim}37°C$, simulating a corrosive environment. After a specified duration, the samples are inspected for corrosion, such as rust or coating degradation, to evaluate their resistance. $NaCl$ is neutral (pH7). Thus, this test was not an accurate simulation of the conditions in the dissolution tester (pH1.2).

The obvious solution to this was to then replace the 5% $NaCl$ with HCl of a specified concentration, e.g., 1-Normal HCl, based on the hypothesis that HCl is a better discriminatory agent than $NaCl$ However, this remains unfeasible as the corrosive nature of HCl could negatively affect the quality and performance of the salt spray fog machine.

The Solution:

In order to combat this issue and prove the hypothesis, a novel testing system centered around the use of HCl was required, which currently does not exist. This would mimic the industrial standard 'salt spray test', while overcoming its limitations (outlined in the previous section) by testing coated mild steel samples with HCl fumes. I aimed to develop this complete testing system by identifying specifications, modeling the system, and building it using various parts available within Electrolab's R&D lab. I also aimed to experimentally test the system to ensure its success.

Specifications:

In order to design the system from scratch, it was necessary to identify specifications and key requirements, which are outlined in Table 1 below with their respective reasons.

Sr. No.	Requirement	Reason
1	Closed System	HCl fumes are poisonous; HCl leaks would lead to unreliable results.
2	Heat Source	HCl must be evaporated for its fumes.
3	Hydrodynamics	The salt spray fog machine achieves hydrodynamics through the movement of fog inside the machine, ensuring circulation, which

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[2] ISO 9227. (n.d.). labomat.eu. https://labomat.eu/gb/faq/613-iso-9227-how-to-conduct-a-salt-spray-test.html

		helps distribute the saline droplets evenly in the machine. In order to mimic this system, hydrodynamics is an essential consideration.
4	Non-corrosive Materials	The testing system cannot be made of mild steel or any other corrosive material, as the paint samples are being tested. If other materials in the system are corrosive, they will react with the HCl fumes instead, making it an unfair test.

Table 1: Specifications

Model:

In order to come up with a base model of the testing system, I had to address each of the aforementioned specifications.

Closed System, Heat Source:

In order to keep the system closed, a desiccator could be used. A desiccator is an airtight container which maintains an atmosphere of low humidity through the use of a suitable drying agent, kept at the bottom of the vessel[3]. However, desiccators are made of plastic or thick glass, which provide unwanted insulation and would disallow heating and evaporation of HCl for its fumes.

Instead, the model was designed with a glass beaker, which allows for heat transfer and thus evaporation of HCl. To ensure the system is closed, the beaker could be capped using an acrylic lid (remains inert through the testing experiment). The system could be further sealed using parafilm, which is a semi-transparent, flexible, and inert self-sealing thermoplastic, often used in similar scientific experiments, models, and investigations.

Hydrodynamics:

Within a beaker, hydrodynamics proved to be difficult to implement. The model was initially designed upon the idea of a 'rotisserie grill', where coated samples were affixed to an acrylic rod by

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[3] Libretexts. (2022, August 15). Proper use of a desiccator. Chemistry LibreTexts. https://chem.libretexts.org/Ancillary_Materials/Demos_Techniques_and_Experiments/General_Lab_Techniques/Proper_Use_of_a_Desiccator

means of perforation (holes). Holes would then be drilled through the beaker for the rod to pass through. The rod would be connected to a motor spun from outside.

However, if the rod were circular, it is likely that the samples inside would not themselves rotate: they would remain stationary while the rod spins alone instead. Thus, this would only be successful if the samples were glued to the rod. However, adding glue was unfeasible given that it was unknown how its chemical composition could affect the results. Instead, a square shaped rod could be used. Yet, the circular holes drilled in the beaker would have to be the length of half the diagonal, which makes sealing the system accurately almost impossible. Additionally, this mechanism required the creation of holes (unless also coated), which adds inaccuracy by exposing parts of the mild steel to HCl fumes directly, thus negating the purpose of the testing process as a whole.

Thus, the model was then based upon the use of another pharmaceutical lab equipment, called the EDI 2I Disintegration Tester, which is a solid state instrument designed for the accurate estimation of disintegration time of tablets as per specific standards[4]. This machine was chosen due to its oscillating basket mechanism, which typically moves a ‘basket rack’ assembly up and down at a fixed speed (using ‘linking rods’), simulating the mechanical action of the digestive system. In this context, the linking rods were directly connected to the samples using the support of metal mesh. Thus, the samples themselves oscillate, ensuring hydrodynamics.

To keep the system sealed, holes (which were the exact radius of the linking rods) were drilled through the acrylic lid. The motor controlling oscillation was outside the beaker, while the samples were inside the beaker, under the lid. Parafilm was used to eliminate any remaining scope for leakage of fumes.

In practical situations, corrosion occurs solely due to exposure of fumes. In order to accelerate the testing process and reduce its duration, the model was developed using an oscillation cycle with dipping and drying, where samples are partially immersed in solution for one hour per day, and are exposed to fumes for the remaining 23 hours.

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[4] Disintegration Test Apparatus – 2901 | ElEctronics India - Manufacturer, supplier and exporter of analytical, scientific laboratory and pharmaceautical instruments in India. (n.d.). Electronics India - Manufacturer, Supplier and Exporter of Analytical, Scientific Laboratory and Pharmaceautical Instruments in India. https://electronicsindia.co.in/product/disintegration-test-apparatus-2901/

Non-corrosive Materials:

As aforementioned, acrylic and parafilm were used when appropriate and possible. For parts of the model where metal was necessary (e.g., linking rods), 316 Stainless Steel (SS316) was used, which is corrosion resistant. Thus, no external materials involved in the testing system would interfere with the process.

Visual Representation:

Fig. 4 below is a labelled diagram illustrating the testing model setup.

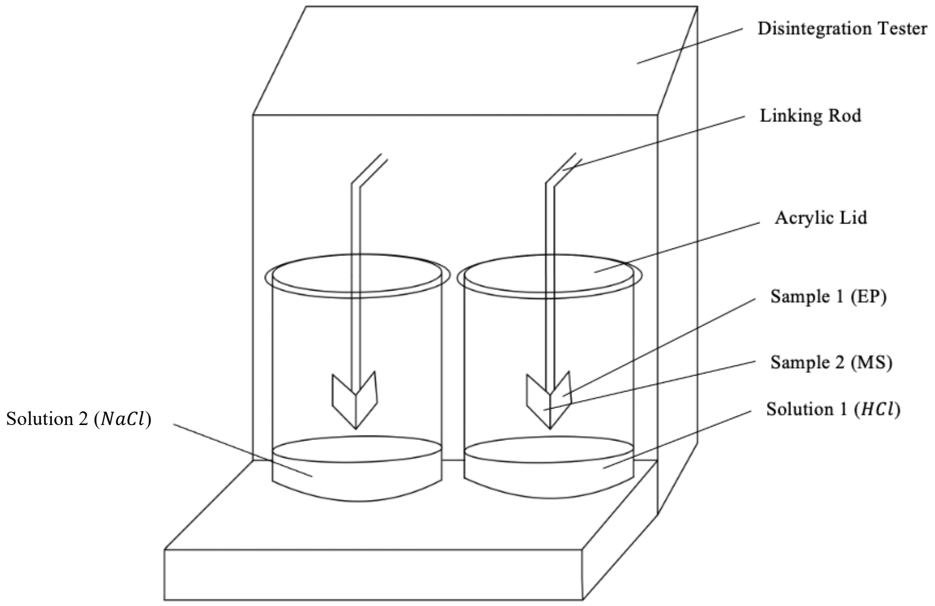

Fig. 4: Diagrammatic Representation of Setup

Section B: Experimental Verification of Testing System

Experimental Outline:

Before this potential new testing system was implemented, it was important to verify that the system would work. Not only would testing the model confirm its own success, but would also be instrumental in confirming the initial hypothesis, that HCl is a better discriminatory agent in testing the efficacy of paint coatings as compared to $NaCl$.

Therefore, two beakers were set up, where one was filled with 5% $NaCl$ solution, and the other with 1-Normal HCl solution, as per the industrial standard ASTM G31-72[5]. In each beaker, 2 samples were tested, one having been coated by the old epoxy-polyester coating, and the second with the newer multi-shield coating. The reason for using two types of coating is to ensure that the system is not biased to any one specific type of coating. This helps validate the consistency and reliability of the testing system, proving results to be representative and widespread.

The experiment was run for a period of 12 days, and the expected results are stated below:

1. In both solutions, the epoxy-polyester coating samples will exhibit rusting (corrosion) earlier than the multi-shield coating samples.
2. Corrosion will occur more rapidly and to a greater extent in the HCl solution, for both samples, compared to their counterparts in the $NaCl$ solution.

Variables:

Independent:

The independent variable is the testing medium. In one system, 5% $NaCl$ solution is used, while in the other, 1-N HCl solution is used. While this will be tested with two different coatings (as outlined above), it is important to note that the coatings are NOT being compared but rather the mediums, to investigate whether or not HCl is a better discriminatory agent.

Dependent:

The dependent variable is the efficiency of the testing system. This will be done by comparing the epoxy-polyester samples in the 2 solutions to each other, and the multi-shield coating samples to each other. In this way, the samples are compared in pairs.

Comparison will be done in multiple ways, listed below:

1. Change in thickness of protective paint coating
2. Change in mass of coated samples
3. Qualitative observations

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[5] Standard practice for laboratory immersion corrosion testing of metals. (n.d.). https://www.astm.org/g0031-72r04.html

Controlled:

In order to keep the comparison fair, certain variables were held constant. These are outlined in Table 2 below, along with their respective reasons.

NOTE: Due to characteristic differences between the two paint coatings, thickness of paint could not be kept constant across *all* 4 samples. Instead, they are kept constant within the coating type (e.g., thickness of both multi-shield coating samples is the same; however, this differs from the thickness of epoxy-polyester coating). This is acceptable as the samples are compared in pairs regardless.

Controlled Variable	**Detail**	
	Epoxy-Polyester Coating Samples	**Multi-Shield Coating Samples**
Thickness of paint	$150\mu m$	$80\mu m$
Substrate	Mild Steel	
Thickness of substrate	$1.2mm$	
Conc. of HCl	1-Normal	
Conc. of $NaCl$	5%	
Dip-Dry cycle	1-23 hours	
Material of lid	Acrylic	
Material of linking rods, mesh	SS316	
Shape of samples	Rectangular	
Size of samples	$45mm$ x $40mm$	
Temperature of solutions	37ºC	
Volume of solutions	$400ml$	
Duration of Experiment	12 days	

Table 2: Controlled Variables

(PTO to proceed)

Method, Data and Quantitative Analysis:

Method and Data:

As aforementioned, solutions were prepared and poured into 2 beakers. To each linking rod, 2 samples were affixed: one epoxy-polyester coating sample and one multi-shield coating sample. The system was setup to mirror the diagram, which was shown in Fig. 4 earlier in the report. Fig. 5 and 6 below show the practical setup before and after sealing respectively. The green color is due to backlight present in the disintegration tester (equipment being used for hydrodynamics).

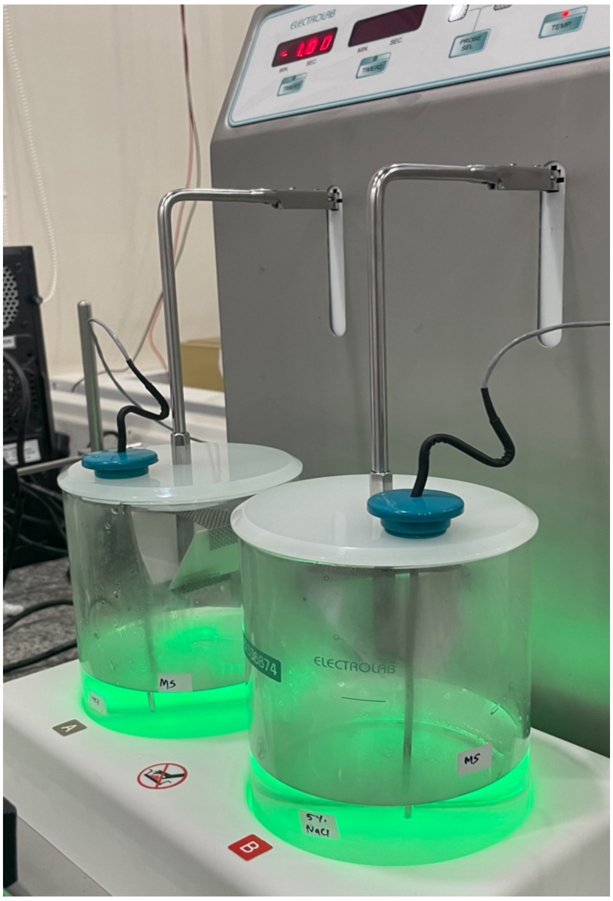

Fig. 5: Practical Setup (before sealing)

Fig. 6: Practical Setup (after sealing)

The experiment was then started and kept ongoing for 12 days. At the end of 12 days, samples were removed from the system and inspected. The masses of the ***samples*** and the thickness of the ***coatings*** were measured in grams (g) and micrometers (μm) respectively. They were compared with their corresponding values (on day 1). These readings are outlined in Table 3 below. They have been rounded to 2 decimal places for simplicity.

Sample No.	Coating	Solution	Coating Thickness (day 1)	Coating Thickness (day 12)	Reduction in Thickness (%)	Sample Mass (day 1)	Sample Mass (day 12)	Reduction in Mass (%)
1	E-P	$NaCl$	150.00	145.00	3.33	16.00	15.23	4.81
2	E-P	HCl	150.00	137.00	8.67	16.00	14.27	10.81
3	M-S	$NaCl$	80.00	79.00	1.25	16.00	15.80	1.25
4	M-S	HCl	80.00	75.00	6.25	16.00	15.01	6.19

Table 3: Data Collected – Mass, Thickness

Analysis and Evaluation:

Fig. 7 and 8 below represent readings from Table 3 graphically. Fig. 7 compares epoxy-polyester samples 1 and 2 in $NaCl$ and HCl. Fig. 8 does the same for multi-shield coating.

Fig. 7: Comparison between epoxy-polyester samples in $NaCl$ and HCl

(PTO to proceed)

Fig. 8: Comparison between multi-shield samples in $NaCl$ *and* HCl

It is evident through the graphs above that HCl has a greater corrosive effect than $NaCl$. With respect to the epoxy-polyester coating, $NaCl$ reduced the coating thickness by 3.33%, while HCl did the same by 8.63%, almost thrice as much. This was further validated by a reduction in mass, where the $NaCl$ sample suffered a 4.81% reduction in mass compared to the HCl sample of 10.81%, which is more than double.

To validate this, the results from multi-shield coating were also considered. With respect to the multi-shield coating, $NaCl$ reduced the coating thickness by 1.25%, in comparison to 6.25% by HCl, a shocking 5 times as much. Further, there was a 1.25% reduction in mass of the sample in $NaCl$ compared to a 6.19% reduction in mass of the sample in HCl. Thus, the trend across both coatings were the same. Using both coatings helps validate that the testing system is impartial and fair.

Across all 4 samples, the multi-shield coated sample kept in $NaCl$ (sample 3) experienced the least corrosion, with only a 1.25% reduction in both coating thickness and mass. The epoxy-polyester coated sample kept in HCl (sample 2) experienced the most corrosion, with an 8.67% reduction in coating thickness and a 10.81% reduction in mass.

Although all four samples experienced corrosion, these results suggest that HCl is a better discriminatory agent than $NaCl$, which is in line with the hypothesis. It also proves that the novel testing method is successful in its job, and that Electrolab can use it to test even their new coating samples, as the method is not partial to the type of coating.

Qualitative Analysis

Results and Observations

Fig. 6 below shows the samples before the experiment started, on day 1. The other two samples had the same appearance.

Fig. 6a: EP Coating

Fig. 6b: MS Coating

Fig. 7 shows the comparison of the epoxy-polyester coating samples in $NaCl$ and HCl (samples 1 and 2) on day 12.

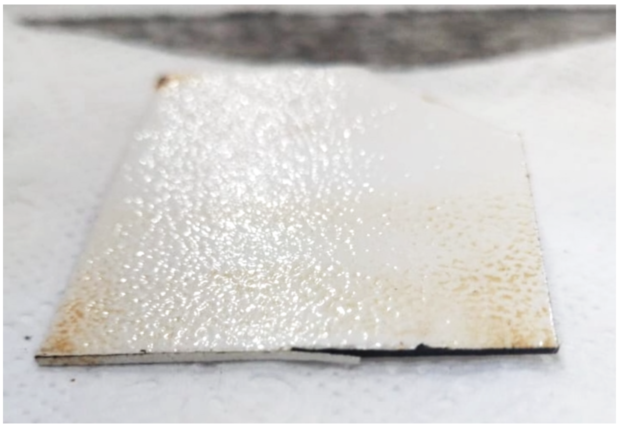

Fig. 7a: EP in $NaCl$ (sample 1)

Fig. 7b: EP in HCl (sample 2)

Fig. 8 shows the comparison of the multi-shield coating samples in $NaCl$ and HCl (samples 3 and 4) on day 12.

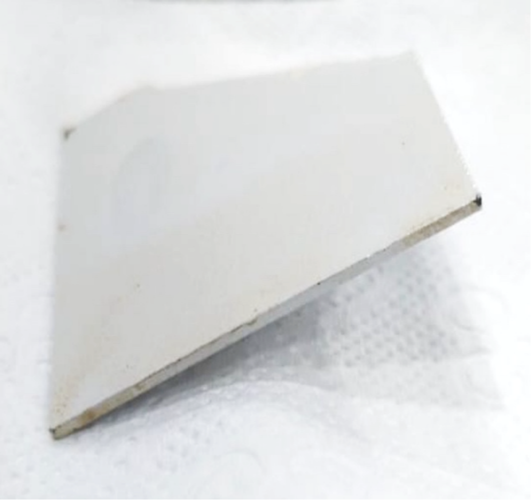

Fig. 8a: MS in $NaCl$ (sample 3)

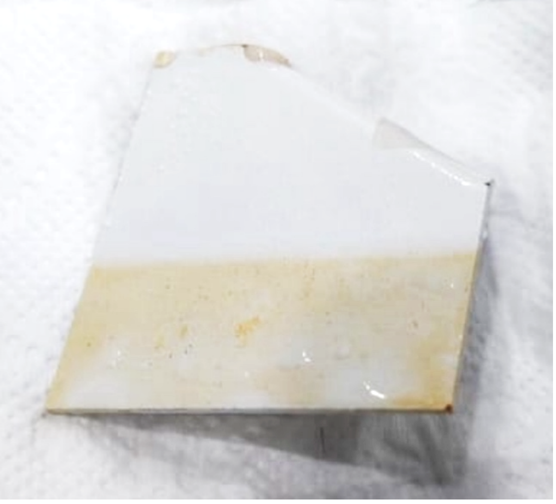

Fig. 8b: MS in HCl (sample 4)

Analysis and Evaluation:

Through the images, it is evident that all 4 samples experienced some degree of corrosion through rusting. However, the multi-shield coated sample kept in $NaCl$ (sample 3) experienced the least corrosion, as only slight rust is visible on the corners. Meanwhile, the epoxy-polyester coated sample kept in HCl (sample 2) experienced the most corrosion and was visibly rusting due to the bright brown-red color on all its edges. This is line with the quantitative data collected.

Moreover, in both coatings, the samples kept in HCl experienced more corrosion as a greater degree of rust is visible on them. Samples in Fig. 7b and Fig. 8b are both significantly more rusted than their counterparts kept in NaCl, which are shown in Fig. 7a and Fig. 8a. This further validates the qualitative data collected, thus proving the hypothesis as correct and the testing system as successful.

Conclusion:

The development of a novel HCl-based corrosion testing system successfully addressed the limitations inherent in traditional salt spray tests for evaluating the durability of protective coatings on dissolution testers. The experiments demonstrated that HCl is a superior discriminatory agent in simulating the corrosive conditions experienced by dissolution testers. This was verified using two coatings, where both experienced greater corrosion and rust in HCl as compared to NaCl, measured by reduction in coating thickness and mass. Two coatings were used to ensure the testing method is not biased.

The new test not only provides crucial insights for Electrolab's future product enhancements but also establishes a methodological framework that can be utilized broadly across industries requiring accurate corrosion testing under acidic conditions. By providing an impartial and effective means of evaluating coating efficacy, this system plays a vital role in enhancing the longevity and performance of pharmaceutical testing equipment.

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