DESIGN FOR SAFE REUSE OF LABWARE: INVESTIGATING METHODS FOR QUALITY ASSURANCE

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ABSTRACT

The problem of plastic waste in research laboratories is a significant one, with an estimated 5.5 million tonnes generated annually worldwide. Reusable labware has the potential to reduce this waste significantly, but the design of such products must take into account quality assurance to guarantee the accuracy of experiments. Insights were gathered through the generation of an overview of the available techniques for verifying labware after use and decontamination. As during different design cycles verification of prototypes is needed, these techniques were evaluated and translated to be applicable in the specific context of a design lab. Therefore, this study presents a protocol which can be used as a verification tool while designing safe, reusable labware for chemical laboratories. This protocol consists of four different steps: (i) visual inspection, (ii) mass & size comparison, (iii) leak test, and (iv) chemical stability test.

Keywords: Quality assurance, Circular economy, Labware, 3D printing, Design process

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1 INTRODUCTION

Laboratories worldwide are using yearly immense amounts of (plastic) consumables during their experiments, such as gloves or pipet tips. This is reflected in the amount of plastic waste generated by research labs worldwide, which is estimated at 5.5 million tonnes per year (Urbina *et al.*, 2015). The initiative #LabWasteDay asked scientists to record the amount of plastic lab waste they generated in a single day, which resulted in an average of 300-400 g of plastic, or 70-100 kg per year for a single scientist (Krause *et al.*, 2020). Research labs are trying to reduce their environmental impact, as they want to join the global movement to a more sustainable society. For example, green chemistry is a rising topic, where solutions such as miniaturisation (Armenta *et al.*, 2019) and the use of alternative less hazardous solvents are common to reduce the amount of (hazardous) waste. As mentioned by (Armenta *et al.*, 2019) plastic waste however, is still often a forgotten problem, with a lack of alternatives both in research and in the market. In the last couple of years, some initiatives have emerged that acknowledge this problem and try to tackle it, for example the University College of London which has the ambition to become a single-use-plastic free campus in 2024, explicitly including their laboratories in this commitment (UCL, 2019). As a large part of this plastic waste consists of consumables, which are only used once before being disposed, reuse of these products will have an immense impact in reducing the amount of waste. For some products, alternatives do exist, but certainly not for all applications. For example, in order to replace single-use plastic containers, reusable glass containers can be used. However, they are not suitable to hold hydrofluoric acid (Hanif *et al.*, 2020).

This research is focussing on how these critical single-use items can be replaced by qualitative alternatives that have a long lifetime and can be reused for multiple usage cycles. Using the Butterfly model of (World Economic Forum *et al.*, 2016) to position our research, we focus on the smallest possible circle within the technical cycle that is aiming for extension of usage through maintenance and reuse to achieve high value retention. This can also be reflected in the popular 9R strategies, focussing on reduce (consuming less materials) and reuse (usage of product for same functionality by same or different users) (Potting *et al.*, 2016).

In laboratories, reuse will come with strict requirements depending on the type of experiment. Contamination between different experiments should be excluded, to guarantee correct results. Furthermore, for example, a scratched surface might also pose a problem for certain analytical techniques such as spectrophotometry (Bishop *et al.*, 2018). Therefore, reuse will only be accepted in this sector if the quality can be assured. Consequently, understanding of quality assurance is essential not only for the end user but also during the development of new reusable labware. A special focus will be needed for the low-tech labware which is often designed as single-use due to its minimal production costs. This study will concentrate on one type of laboratory, specifically the chemical laboratory. Consequently, the sterility requirements are typically less strict than, for instance, in a medical laboratory, but the labware must be resistant to harsh chemicals. We consider the criticalities in chemical labs as a reference case for criticalities in other sectors such as reusable food packaging, where contamination can also have a significant impact on the preservation of the food.

This publication is part of a bigger interdisciplinary research project in which a bridge is made between the chemistry community and the design community with the intend to achieve knowledge for the design of qualitative safe reusable labware and consumables. In the project, focus is put on the usage context (lab context) (Van Loon *et al.*, 2020), the users (lab workers) (publication in review), the cleaning activities (Van Loon *et al.*, 2021). The aim of the research described in this publication is to understand how reusability and the remaining quality can be evaluated in order to ensure quality, understanding of optimal cleaning and resetting for reuse is important to ensure full cleanliness, sometimes even sterility, and optimal preservation of both shape and material conditions. Consequently, research question 1 was formulated as:

RQ1. What techniques are available that can be used to evaluate the safety of reusable products?

As we aim to translate these insights into relevant design insights, the second part of the research focusses on the increasing usage of additive manufactured labware products that are used in lab contexts in order to enable specific tailormade tests. The low quantity and consequently high cost per lab product increase the need for reusability. Understanding how to optimally design these products as well as how pretesting can be done during the design process could increase the effective reusability of

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products. Consequently, RQ2. How should these existing techniques be adjusted to be used in a design environment?
The results of this paper bridge the domains of both the sustainable design SIG with the Design for Additive Manufacturing SIG within the Design Society.

2 MATERIALS AND METHODS

2.1 Overview of currently used techniques for verification of reusable labware (RQ1)

First, the state of the art was explored in literature and textbooks, using scientific search engines, more specifically, Google Scholar, Web of Science and Scopus. Search terms consisting of various combinations of two or more of the following keywords were used: "labware", "lab equipment", "evaluation", "verification" "decontamination", "cleaning", "reuse", "deformation", "temperature", "moisture", "chemical", "resistance". No specific requirements were set for year of publication. This resulted after a first selection based upon the titles and a review of citations in 45 articles and 34 textbooks or book chapters. Due to the limited number of articles all of them were analysed for relevance. More general information on techniques was found in the selection of textbooks. In addition, ISO standards databases were consulted to find relevant test methods.

2.2 Translating verification techniques to design environment (RQ2)

This was followed by a second consultation of the same scientific search engines for the second part of the article, this time focusing on verification methods for 3D-printed labware. Here, search terms consisting of various combinations consisting of two or more of the following keywords were used: "rapid prototyping", "additive manufacturing", "3D printing", "labware", "lab equipment", "evaluation", "verification", "chemistry", "decontamination", "cleaning", "deformation", "temperature", "moisture", "chemical", "resistance". Publications were filtered based on year of publication, as 3D printing is a rather new technology. All publications, published after 2012 were considered. This search resulted in 61 research articles, in addition 1 textbook was consulted. This info was reviewed together with design experts in order to select or translate the current techniques into techniques that can be applied in the design environment.

2.3 Graphical flowchart of article structure

To explain the structure of the research method a graphical reasoning model is provided in Figure 1.

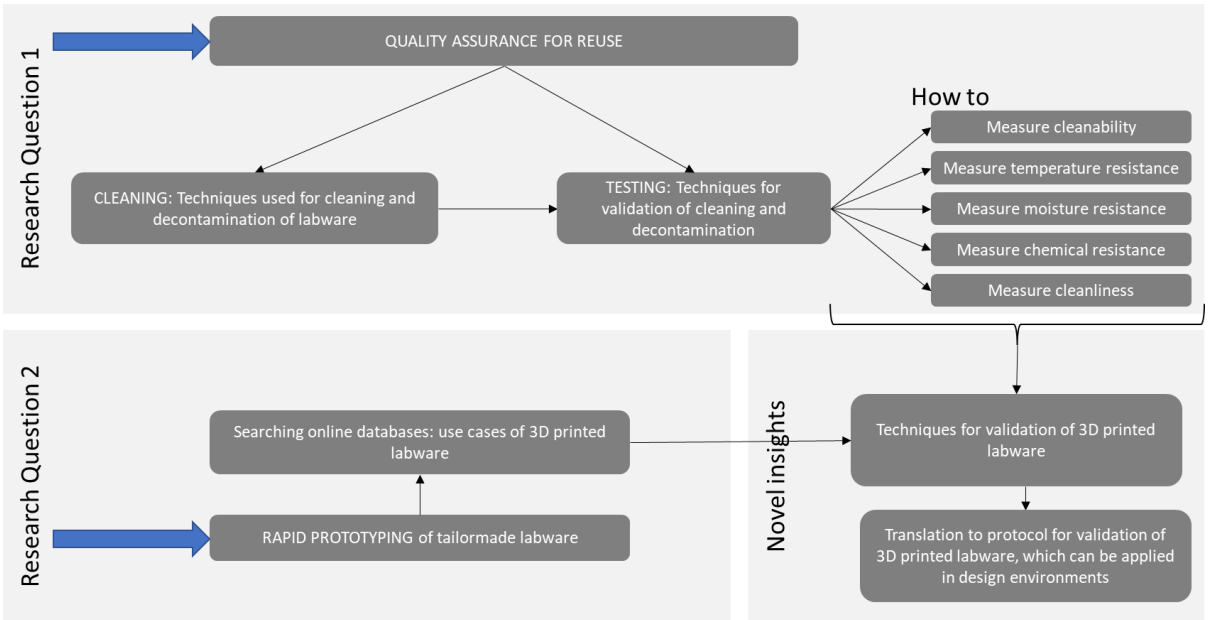

Figure 1: Graphical flowchart of article structure

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3 RESULTS

3.1 Techniques for cleaning and decontamination

To reuse labware, the most important step is cleaning. Often, additional decontamination or sterilisation is needed, depending on the application. The products should be able to resist the often harsh conditions of these techniques. Therefore, an analysis of the existing techniques and their conditions is necessary.

According to the (World Health Organization, 2020) a product can be considered clean when it is visually free of soil and it is below specified levels of analytes. Cleaning can be performed manually by scrubbing with warm water, or with the use of a laboratory dishwasher (World Health Organization, 2020). Frequently, a cleaning agent, such as a soda or soap solution or another commercial preparation, is added to enhance the effectiveness of the process (World Health Organization, 2020). It is recommended to rinse labware with water after cleaning it with detergent to ensure that all traces of detergent have been removed (Bishop *et al.*, 2018; Seidman *et al.*, 2021). The compatibility of detergents and wash cycle temperatures should be checked with the manufacturer recommendations (Bishop *et al.*, 2018). The use of organic solvents might be necessary when detergents are not able to clean laboratory glassware, the most used types are acetone and ethanol (Isac-García et al., 2015). The use of organic solvents on plastic should be wisely considered as not all types of plastic will be compatible with all types of solvents (Bishop *et al.*, 2018). In addition, ethanol is also a type of chemical decontamination within the group of the alcohols. They are effective against a wide range of bacteria and enveloped viruses (World Health Organization, 2020). Alcohols tend to evaporate quickly, rendering them ideal for surface disinfection, but also decreases the possible time of exposure (World Health Organization, 2020).

Thermal decontamination methods are the most frequently used ones within laboratories, two types do exist: (i) moist heat decontamination, (ii) dry heat decontamination. Moist heat or steam sterilisation, which is commonly carried out with an autoclave, utilises thermal energy to sterilise an object. By means of saturated steam under pressure, this thermal energy permeates the item. (Tranquillo *et al.*, 2022; Wendt *et al.*, 2015). There are two standard protocols, one using 121 °C for 15 min and a second using 132 °C for 4 min (Wendt *et al.*, 2015). For heat-sensitive materials, alternatives should be used.

With the use of dry heat as a decontamination method, higher temperatures are required to achieve the same result. The exterior surface of an object absorbs and transfers dry heat throughout the entire object. The most common time-temperature parameters for dry heat sterilisation are 170 °C for 60 min, 160 °C for 120 min, and 150 °C for 150 min (Sastri, 2022). As dry heat requires higher temperature and longer exposure time, it is generally only used for materials which cannot withstand steam or which cannot be penetrated by it (Zhou and Breyen, 2013).

3.2 Measuring cleanability

All of these methods of decontamination may have some impact on the final product. To allow reuse with the same level of confidence in the product's quality, the product should be able to continuously withstand these severe conditions over time. Based on the above cleaning and decontamination techniques, a number of assessable factors emerge. In the first place, while applying a thermal form of decontamination, the product should be able to withstand these high temperatures. Material selection and wall thickness will be the most relevant factors in this instance. The second will be moisture resistance, which means that in the event of moist heat decontamination, the moisture cannot be absorbed and trapped within the product. The third one should be the chemical compatibility with potential solvents used to clean laboratory equipment; if certain solvents are incompatible, this should be indicated clearly. Under normal circumstances, these factors will be analysed in specialised laboratories equipped with a range of methods.

3.2.1 Measuring temperature resistance

Based on available material characteristics, most of the time measurement is not essential. However, if the limits of these characteristics are touched or/and combination of factors might shift these limits, testing is applicable. The most common effect of failing temperature resistance is deformation, including melting. Larger deformations can be observed visually, but smaller scale one should be

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examined in other ways. The relevant ISO standard in this instance is ISO 3146, which describes a method for determining the melting temperature of plastics by visually observing the point at which the test specimen changes shape while being gradually heated in a capillary tube (International Organization for Standardization, 2022a).
These deformations can influence the leak tightness of products and the accuracy for measurement purposes. One of the most used methods to analyse temperature resistance is Differential Scanning Calorimetry, measuring exothermic and endothermic changes (Swallowe, 1999), these changes happen at phase transitions due to changes in molecule mobility. In order to measure the airtightness, there are three standard tests: (i) measuring the flow of helium through an object (ii) measuring pressure decay over time, or (iii) immersing a product in liquid and measuring the leakage flow by the volume and intensity of bubbles escaping (Vinogradov *et al*., 2016). For each of these methods, an acceptable level of leakage should be determined beforehand.

3.2.2 Measuring moisture resistance

High humidity can also lead to melting, and thus deformation, mainly caused by the temperature of the moist. But some materials, such as polyamide, are sensitive to high humidity themselves, and in these cases this might lead to moisture swelling due to absorbance (Zhou and Breyen, 2013). In the case of (partly) metal products, moisture might lead to oxidation as well (Zhou and Breyen, 2013). ISO 62 defines the method to analyse water absorption of plastic. The reference test in this case is an exposure to 50% relative humidity at 23.0 °C for 24h whereby the sample is weighted before and after the exposure using a balance with an accuracy of 0.1 mg (International Organization for Standardization, 2008).

3.2.3 Measuring chemical compatibility

The lack of resistance to chemicals will intensify the process of degradation, in case of degradation the molecular weight will be reduced and therefore the mechanical properties will change. To some degree, these changes can be observed visually, for example, a change in colour or texture, or by mechanical testing to verify changes in physical properties (ASM International, 2003). But to observe changes in molecular microstructures, spectroscopic techniques will be necessary (ASM International, 2003) .
A relevant ISO standard which performs equal testing is the DIN ISO 175, whereas samples of plastic are immersed for up to 90 days in a set of chemicals and changes in dimensions and weight are measured as well as changes in appearance are evaluated (International Organization for Standardization, 2010). In addition, tensile tests are conducted on the dried samples to observe changes in mechanical strength (International Organization for Standardization, 2010).

3.3 Measuring cleanliness

During usage and cleaning, chemicals and substances may be left as residue on product surfaces. To assess the products' cleanliness, a variety of techniques are available based on the type of contaminant and the precise measurement objective (Kohli, 2012). The ISO standard 14644-10 includes a list of techniques, such as Fourier-transform infrared spectroscopy or X-ray photo electron spectroscopy, suitable to analyse contaminants on surfaces (International Organization for Standardization, 2022b). More precisely, ellipsometry or X-ray techniques could be used to determine the thickness of a film layer; microscopy or optical particle counters could be used to determine the number and size of particles remaining on a surface; and spectroscopy would be the most appropriate technique for determining the distribution of organic contaminants on a surface (Kohli, 2012). All of these approaches must be carried out by highly qualified and trained laboratory employees implementing the appropriate sample preparation.

4 REASONING: HACKING THE TECHNIQUES TO APPLY IN DESIGN ENVIRONMENTS

Understanding the existing techniques is interesting, however, in order to support future designers, it is essential to translate the applicability and relevance of each technique in the context of a new designed reusable product.

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4.1 The designer approach

During the design process, intermediate verification is a crucial step for iterating through various cycles. Prototypes are the most accurate representation of the final product for the purpose of verifying the design from both a user as well as a technical perspective. In recent years, 3D printing has become one of the most important techniques for rapid prototyping in product design (Medellin-Castillo and Zaragoza-Siqueiros, 2019; Nazir *et al.*, 2021). For example in design firms in the UK, 3D printing is now a common practice within the iterative design cycle to achieve high fidelity prototypes throughout the whole cycle (Goudswaard *et al.*, 2021). One of the primary reasons for this is the ability of experienced prototypers to produce highly representative results with characteristics similar to those of the final product. This is not possible for instance, with cardboard models. The most important aspects of prototyping as part of the design process are (i) the method's cost, (ii) the required time, and (iii) the skills required. In sum, 3D printing is ideally suited to the skill set of a product designer, with 3D digital drawing/Computer Aided Design (CAD) being the skill with the highest barrier to entry. In comparison to other techniques that require the acquisition of new skills, 3D printing requires a minimal investment from product designers as they are literate with CAD (Goudswaard *et al.*, 2021). A high-end FDM (Fused deposition modelling) 3D printing machine can be acquired for less than one thousand euros (Prusa Research, 2022). Depending on the size of the prototypes, the majority of objects can be manufactured overnight.

It should be noted that substantial differences appear between the characteristics of the 3D-printed prototype and the final design in terms of materials, surface finishing and producibility. Consequently, verification of reusability, especially cleanability and cleanliness, cannot easily be done using prototypes.

Furthermore, during the process of verification while rapid prototyping, it is difficult to apply complicated lab techniques. These lab techniques to measure influences on products require a very different set of skills than those of a product designer. Having to go to an external lab every time and start performing measurements with the help of other experts is a very delaying process in this verification. Therefore, it is necessary to explore methods to perform these verifications in the environment of a product design environment.

4.2 Exploring current use of 3D-printed prototypes for (reusable) labware analysis

In order to achieve a good reasoning on the designers' skills as well as usage of 3D prototypes, we based upon existing research that uses 3D prints as actual labware within chemical experiments. The first major issue for lab work is to point out that "fresh" 3D prints are not sterile nor clean. This has been demonstrated by (Diep *et al.*, 2022), by means of a basic experiment in which 3D prints were, immediately after fabrication, placed in a broth and showed growth of organisms; this was not the case for 3D prints that were autoclaved after production. Sterility is certainly not necessary for every application, but it should be known that even before the first usage, cleaning of 3D-printed products is essential.

A first type of evaluation is the exclusion of **deformation**, this might be a visual check in case of large deformation. Small deformations can be measured with a profile projector device and compared to the original profile of the product before decontamination (Boursier *et al.*, 2018). A more affordable method to measure deformations is with the use of a (digital) calliper with a high accuracy (Heikkinen *et al.*, 2018), or by the use of photogrammetry to compare the profile of two products with the use of a fixed setup and image overlaying. This is a type of verification that can be conducted in a design environment. The utilisation of a precision scale and a calliper will be the most practical method. As with all the subsequent types of verification, it should be noted that the production technique of the prototype will differ from that of the final product, which can affect the outcome. This also applies to the materials, as only thermoplastics can be 3D-printed. A second issue that may arise with 3D printing is a lack of **leak tightness**. Using the appropriate parameters in the 3D printing software will affect this property. Leak tightness should be evaluated on both the original and decontaminated components to identify any changes. Leakage may result from the deformation or extraction of material by solvents. A relative easy setup was made by (Gordeev *et al.*, 2018), where the 3D-printed labware was connected to an air compressor and pressurised to 1 mbar while submerged in water to detect whether air bubbles escape. If air bubbles were visible, this indicates that air can permeate the wall and that the product is not airtight. This type of verification should be possible within a design

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environment, as it only requires low-tech and low-cost investments, namely, the purchase of an air compressor. Connecting the prototype to the air compressor is the primary challenge; the degree of difficulty will depend on the prototype's size and shape. For prototypes with cylindrical openings, gas-tight rubber tube caps with openings for tubing can be used.

A final group of tests is to evaluate the **compatibility with common chemicals** used within chemistry labs. To evaluate the resistance to solvents, (Gordeev *et al.*, 2016) used 3D-printed test tubes filled with solvents and stirred for 1 hour at both 20 °C and 50 °C. The quantity of plastic extracted by a solvent was then determined by transferring the solvent into a flask, evaporating the solvent, and then weighing the flask. Most of the other research focused on the absorbance of chemicals instead of the extraction of plastic to show decreased or insufficient chemical resistance. In these cases, representative samples of 3D-printed labware made with the same technique and material, were submerged in commonly used chemicals for a certain amount of time. (Heikkinen *et al.*, 2018) evaluated mass and dimension changes after one week of immersion using a digital calliper and a precision scale. Prior to measurement, all submerged components were surface-dried with an N2 gun (Heikkinen *et al.*, 2018). Similar procedures were used by (Raddatz et al., 2017) to evaluate chemical stability; however, the 2-hour immersion, in this case isopropanol and acetone, was considerably shorter. Prior to and following immersion, samples were dried at 70 °C for 60 min and weighed, an increase in mass would indicate the absorption of chemicals (Raddatz *et al.*, 2017). (Popescu *et al.*, 2021) immersed 3D-printed test cubes in two different disinfectants and analysed the absorption through mass gain over time by measuring them after drying with a cloth at 15 different time intervals over a one-week period. A more visual method was developed by (Erokhin *et al.*, 2019), in which a 3D-printed cylindrical sample is submerged for 20 h in 12 distinct solvents. In addition, a metal or glass bead was placed on top of the sample to serve as a load indicator; without a load, a product may appear intact while it is actually losing its mechanical strength (Erokhin *et al.*, 2019; Gordeev and Ananikov, 2020). Deformation was documented by taking images every 10 min (Gordeev and Ananikov, 2020). An additional step was taken by (Heikkinen *et al.*, 2018), after measuring weight or mass change, the samples were tested using differential scanning calorimetry (DSC) to evaluate changes in the crystallinity, glass transition and melting temperatures of the polymers. In the setting of a design environment, the approach described in the protocol of (Erokhin *et al.*, 2019), will be the most relevant, making use of two commonly available and widely used compounds, namely, acetone and ethanol.

4.3 Translation towards verification in a design environment

In the case of a design environment, further verifications are not opportune at this stage of the process. Such as verifying cleanliness, here the primary assumption is the optimal operation of the lab dishwasher, if it is shown that the product itself has not been impacted by this technique, it can be assumed at this stage that the goal of removing contamination to the desired level has been achieved. Obviously, if visible residues are still present, the product will fail the initial visual inspection and be rejected. Similarly, for the optional follow-up step of sterilisation, it can be assumed at this stage that the product has reached the desired level of sterility if the sterilisation procedure has been performed correctly; here, the available and proven sterility indicators, such as discolouring strips, can be used.

Based on the previous insights, three different types of failures after decontamination could be evaluated. These include (i) deformations, (ii) leak tightness, and (iii) chemical resistance/stability. To be able to evaluate these types of failures inside a design environment, a new protocol is needed. This protocol should include verifications on three types of failures in a logical sequence that minimises the amount of time required and prioritises non-destructive verifications over potentially destructive ones. This protocol is represented in Figure 2, and includes four steps to follow, starting with a visual check, followed by a mass and size comparison, a leak test, and a chemical stability test, before a prototype can be marked as acceptable for further proceeding in the (re)design cycle.

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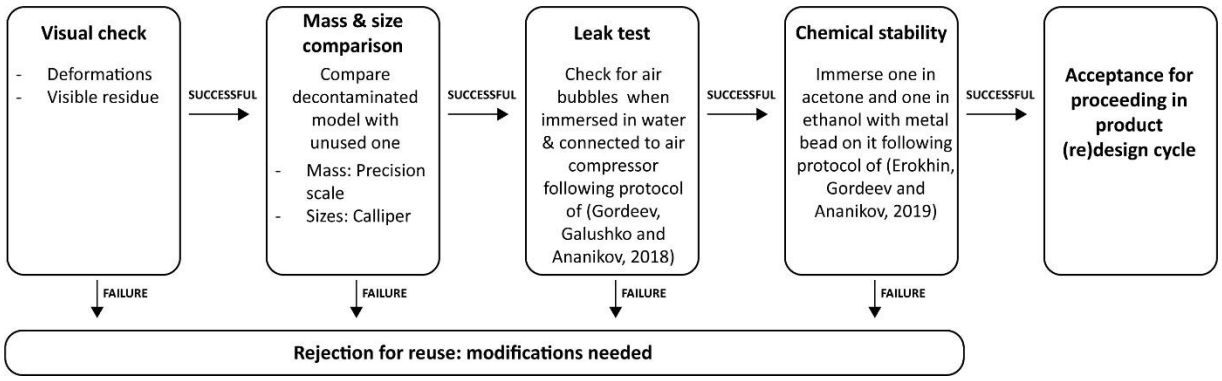

Figure 2. Protocol to evaluate prototypes of reusable labware

5 CONCLUSIONS

Laboratories contribute significantly to the production of plastic waste due to their usage of single-use labware. This research investigated the different techniques that can help in the design of successful reusable labware products. The availability of a clear and straightforward testing protocol for labware in a design environment could be a first substantial step towards making research into the reuse of these products more practical. Modifications to the design, as well as, for example, tools that ease the decontamination process for the user, can be verified more efficiently. This protocol provides a useful starting point, but its effectiveness should be evaluated through further case studies in order to optimise and potentially adapt it if necessary. The availability of this protocol will make the transition to more sustainable labs just one more step easier, as today there is often a lack of alternatives to the labware.

The second phase will consist of convincing labware manufacturers to adopt reusable alternatives. On the one hand, laboratories will demand this as a result of increased awareness. On the other hand, this is consistent with evolving laws that tend to prohibit single-use products. To be prepared for the future, producers should prepare themselves for this changing environment. Consequently, they can capitalize on brand-new business opportunities, such as offering labware washing in large quantities. In the context of this article, prototypes that are a representation of the final product are still being used, this involves 3D printing techniques, which are different from the final production processes for labware. These influences, such as surface finishing, should not be overlooked either. In addition, not all materials are able to be translated into a 3D-printed prototype, only thermoplastic materials can be used. The use of a different material than the final product will have a substantial impact on the results of the measurements. When progressing further in the design process, prototypes that are even more representative should be produced, and efforts should be made to verify cleanliness. This will necessitate collaboration with an external, specialised laboratory with a wide range of available analysis techniques.

Future research is needed to understand optimal the cleaning conditions both through the cleaning context as well as through the internal shape of products. This will decrease the contamination risk through reducing the likelihood of substances to remain in the products. Furthermore, it should be noted that no business not impact-related research is done, these insights are in essence the starting point to step into the transition towards reusable product usage.

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