# PARTICLE REINFORCED POLYMER COMPOSITE'S STAIN RESISTANCE FACTORS

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**Abstract**
*The matrix of the particulate composite is unsaturated polyester resin that is reinforced with alumina trihydrate particles. The goal of the study was to evaluate particle reinforced composite's stain resistance and to investigate the factors that influence it. In order to evaluate the influence of individual components test specimens with varying composition were fabricated. The influence of surface treatment and surface roughness to stain resistance was the other major factor under study. Test specimens were moulded with vacuum assisted extruder. The concentration of polymerization initiators methyl ethyl ketone peroxide and cobalt compound had considerable effect on increasing the stain resistance. Specimens with lower surface roughness had better cleanability.*

## 1 Introduction

Thermosetting resins are reinforced with continuous or short fibres or particles to form a composite material. The most common filler materials for producing non-gel coated particulate composite products are alumina trihydrate (ATH), quartz, resin chips and recycled thermoplastics [1], [2]. The addition of particulate reinforcement increases the stiffness, reduces the shrinkage and thermal expansion, lowers the cost and modifies the rheological and physical properties of the composite material [1], [3]. Unsaturated polyester resin together with alumina trihydrate forms a tough, rigid and hard composite [4].
Current study investigates particulate composite that is used in building and construction industry. More precisely the material is used for fabricating laboratory and culinary bench tops, vanity tops and sanitary ware like washbasins, shower trays and bathtubs.
Stain resistance is one of the top priorities when developing material for such applications. Stain resistance depends from the chemical composition of the components, curing and post-curing of the material and surface treatment of the product [5], [6].

## 2 Materials and methods

*2.1. Sample preparation*

In order to get adequate results the material samples were fabricated in the production facility. The test slabs measured 400 × 1000 × 10 mm. The slabs were casted with a closed mould with a special vacuum assisted casting machine. A closed mould guarantees equal thickness

and flatness of the slab. That is necessary to get test specimens like specified in testing standards. Vacuum chamber of the machine removes air from the casting dispersion and helps to achieve non-porous material. The proportion of filler and other components is controlled by the machine.

For the fabrication of specimens an unsaturated polyester casting resin based on isophthalic acid and neopentyl glycol was used. The resin is developed to produce non-gel coated products and contains methyl methacrylate. It is pre-accelerated, medium reactive, low viscosity resin.

For curing a peroxide mixture based on methyl ethyl ketone peroxide was used. The peroxide is intended for room temperature cure of unsaturated polyester resins and it has low peak exotherm and good final cure.

ATH with fine particle size was used as filler material.

Preliminary cure of the composite was done at room temperature (23 °C ±2 °C) for 12 h. That was followed by post cure at (40 °C ±2 °C) for 16 h.

Five different test slabs (Table 1) were casted to determine the influence of individual components.

| **Materials** | **MA-1 (wt.%)** | **MA-2 (wt.%)** | **MA-3 (wt.%)** | **MA-4 (wt.%)** | **MA-5 (wt.%)** |
|---|---|---|---|---|---|
| Resin | 45 | 35 | 35 | 35 | 35 |
| Filler | 55 | 65 | 65 | 65 | 65 |
| Catalyst | 1 | 1 | 1 | 2 | 2 |
| Accelerator | 0 | 0 | 1 | 0 | 1 |

**Table 1.** Composition of test slabs under observation

*2.3. Surface treatment*

In order to determine the influence of surface treatment and surface roughness to the stain resistance all the slabs were prepared with 4 different surface finishes. First quarter of the slab was untreated mould surface. Second quarter was polished. Third quarter was sanded with P1500 sandpaper and last quarter was sanded with P240 sandpaper.

*2.3. Staining*

The staining and cleaning procedure was carried out according to ISO 19712 – 3 Method B. The test specimens were left on the surface of the material for a period of 16 h. The surface was treated with 15 different staining agents.

## 3 Results and discussion

*3.1 Resistance to stains*

The amount of staining was evaluated after each cleaning step:

1. Washing with water and sponge.
2. Washing with non-abrasive cleaner and sponge.
3. Cleaning with abrasive cleaner and stiff bristle brush.
4. Cleaning with cotton ball saturated with ethanol.
5. Cleaning with cotton ball saturated with bleach.

Each stain was given a rating 0-4 according to the step when it was removed or a rating 5 if it remained visible after step 5. The staining of the test specimen is reported as the sum of the ratings of the 15 agents (Table 2). A higher value indicates more severe staining.

| Surface treatment | MA-1 | MA-2 | MA-3 | MA-4 | MA-5 |
|---|---|---|---|---|---|
| Untreated | 42 | 44 | 50 | 57 | 54 |
| Polished | 62 | 63 | 57 | 62 | 65 |
| P1500 | 58 | 63 | 62 | 66 | 63 |
| P240 | 64 | 63 | 46 | 56 | 57 |

**Table 2.** Staining test results

*3.2 Stain resistance and chemical composition*

*3.2.1 Resin and filler*

Unsaturated polyester resin modified with acrylic is the best compromise between cost and properties for this kind of application. It has natural resistance to household chemicals and stains. Because of low viscosities that allow high filler content and easy casting it is the most common resin in manufacturing of engineered stone products [7].

ATH is used in products where pure white tone is desirable. Alumina trihydrate is a non-toxic, non-corrosive, non-cancerogenic, odourless, flame retardant filler material. It is a mineral derived from bauxite [8].

In order to evaluate the influence of resin wt.% to cleanability one has to compare compositions MA-1 and MA-2. Higher resin content has a slight positive effect on cleanability. Nevertheless, higher resin content increases the net value of the material and it is not reasoned.

*3.2.2 Catalyst and accelerator*

The curing of polyester resin systems is carried out at elevated temperatures of about 100°C or at room temperature with polymerisation initiators. Methyl ethyl ketone peroxide or cyclohexanone peroxide is used for room temperature curing in conjunction with a cobalt compound such as a naphthenate, octoate or other organic solvent-soluble soap.

The peroxides are referred to as "catalysts" and the cobalt compound as an "accelerator". Peroxides activity varies according to the composition of the mixture. Peroxide content is usually 0.8-2.5% by weight. The cobalt solution is normally used in quantities of 0.5-4.0% based on the polyester. Resin systems are often pre accelerated and on site is added only catalyst [9].

The influence of catalyst was evaluated by casting test specimen with 1 wt.% of catalyst and another with 2 wt.% of catalyst. The staining test results do not favour neither one, because the test evaluates if a stain is evident or not. The test does not give any feedback how severe the remaining stain is. When the two specimens were compared side by side, it was evident that the remaining stains on the MA-2 were much more severe than the stains on MA-4. A higher catalyst concentration in MA-4 improves the polymerisation process and boosts the cross-linking. A more linear polymer is chemically more vulnerable than a cross-linked polymer. A suitable solvent that is able to constitute secondary bonds with polymers chains replaces interchain secondary bonds and dissolves linear and branched polymers [5].

The test results and side by side comparison of MA-2 and MA-3 show that the presence of accelerator also improves the stain resistance. Some stains remain on the surface after testing but are much lighter when compared to the test specimen without cobalt compound.

Final test specimen MA-5 was casted with 2 wt.% of catalyst and 1 wt.% of accelerator. There was not any obvious improvement in stain resistance when compared to MA-3 and MA-4.

*3.3 Stain resistance and surface treatment*

Particulate composite without gel coat surface has matt or semi-gloss surface after demoulding. To enhance the aesthetical appearance of products the surface is sanded or polished. This causes a relevant material removal ranging from 0.2 to 0.8 mm. Moreover, the surface is expected to be repairable and workable.

It has been observed with porcelain stoneware tiles that grinding, sanding and polishing operations induce a significant degradation of the superficial characteristics, due to both the formation of cracks and flaws produced by the machining procedure and to the opening of closed pores occurring into the ceramic body. The resistance to stains is to a large extent related to the "microstructural quality" of the tile surface, thus amount, size and morphology of defects (e.g. pores, cracks, grooves) [6], [10], [11].

One of the goals was to evaluate if there are present any closed pores in the surface layer of the composite. In addition, if sanding or polishing open these up. Second purpose of the test was to evaluate how different surface roughness influences the staining.

The untreated surface has the best staining test results. The rest of the surfaces have more or less same results. Unfortunately the results are misleading, because the test does not evaluate the severity of the remaining marks. For example, the remaining stains were lighter on the polished surface than on the untreated surface. Moreover, the polished surface had poor test results because it is easy to spot any defects from a glossy surface. An objective evaluation would be that the easiest to clean was polished surface and the remaining stains were the lightest. The surface treated with P1500 sand paper and untreated surface had similar cleanability rating. The most stubborn to cleaning was surface treated with P240 sand paper. The advantage of untreated and P240 surface was their matt appearance that did not reveal stains that affect the gloss.

## 4 Conclusions

The research was carried out to study the effect of different components and surface treatments to the stain resistance properties of particle reinforced composite. Experimental part included fabrication of material specimens, sanding and polishing operation, stain and cleanability test and evaluation of stain resistance properties.

The test data acquired showed that increased catalyst wt.% and presence of additional accelerator increases the cross link density and thus chemical and stain resistance properties. Smaller surface roughness has favourable effect on stain resistance. Best results were obtained with polished surface and worst with sanding with P240 abrasive paper. There was no evidence of closed pores. The polishing or sanding processes did not lower the stain resistance compared to the untreated surface.

The study showed that the ISO 19712-3 stain resistance test has some weak points and is suitable for preliminary studies. Further studies with spectrophotometer and SEM should be carried out in order to obtain accurate evaluation of colour changes and porosity. The test results obtained betoken a prospect for the tested material to be used commercially as a material for laboratory, culinary, marine or agricultural products.

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