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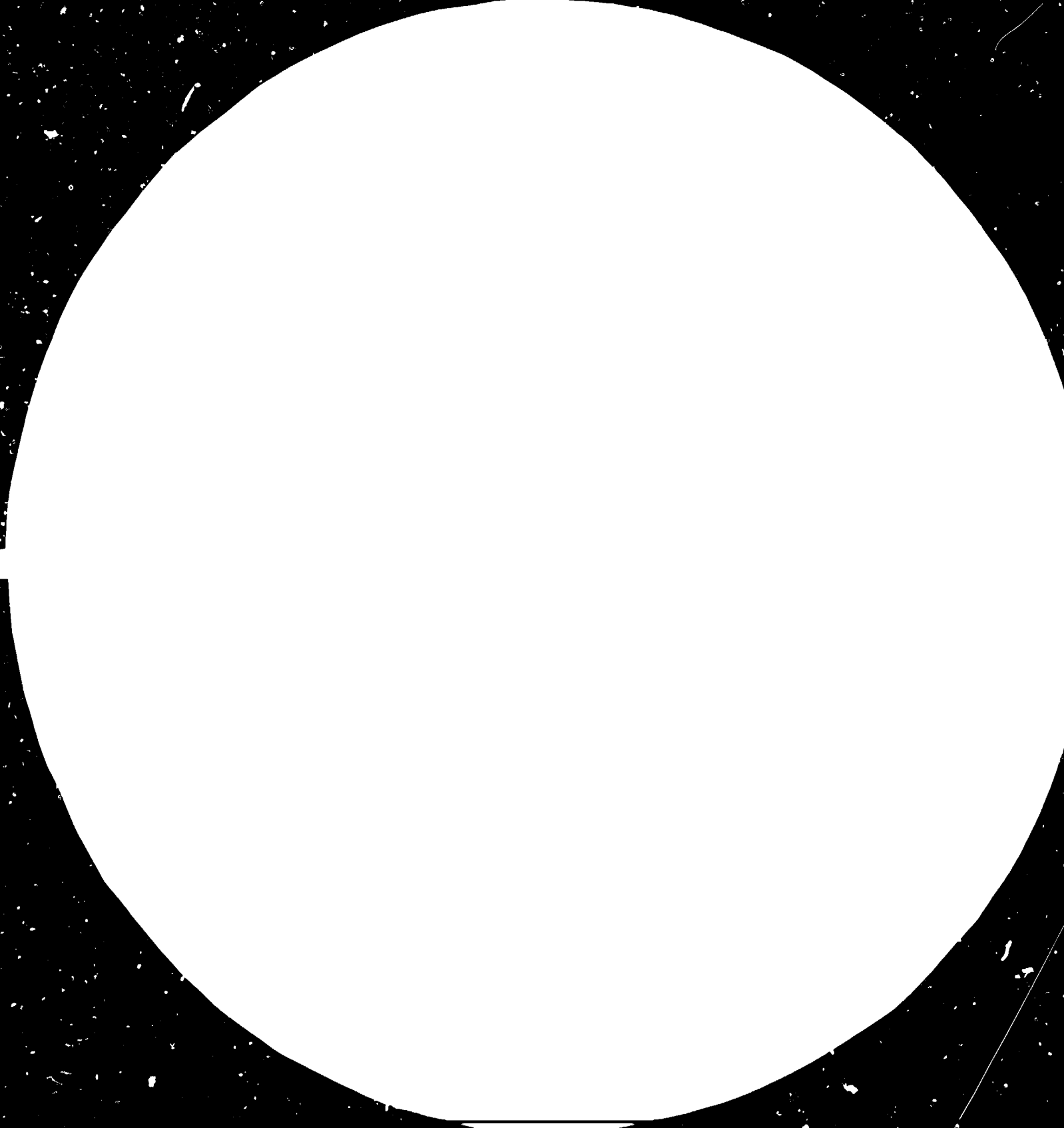
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# 13040

UNITED NATIONS  
INDUSTRIAL DEVELOPMENT ORGANIZATION

Distr.  
LIMITED  
UNIDO/IO.563  
1 November 1983  
ENGLISH

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CERAMIC GLAZES .

Basic Information\* .

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ABSTRACT

This concise publication deals with ceramic glazes which enrich ceramic products not only with impermeability and higher strength but make them also brilliant, impressively coloured and easy to clean.

The applied raw materials and other glaze components are described, glaze compositions are reviewed and principles of coloured glazes explained.

A list of references to publications on glazes is convenient for readers who are interested in a deeper study.

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## I. INTRODUCTION

The industrial production of glazed ceramic products enjoys a long tradition. It is based not only on a series of local clays, kaolins, feldspars and further raw materials but also on the ability of technicians to provide ceramic products with surface dressing upgrading their aesthetic appearance and use value. Glazes rank among the most important substances for surface treatment of products of fine and structural ceramics.

The contemporary phase of development is characterized by increased rate of construction development in developing countries. Structural ceramics as one of the factors of material and technical basis of construction industry is being extended. The increasing standard of living initiates imports of fine ceramics to developing countries although nearly all these countries are rich in ceramic raw materials and could produce these wares themselves.

This publication has been prepared for technicians employed in ceramic plants in developing countries and for trainees in the art of ceramic production. It delivers technical information on fundamental raw materials, glaze composition and selection, glazing methods and defects occurring in practice. The publication involves the knowledge of the author gained from basic and applied research of Czechoslovak and foreign development and research centres. The paper should also serve as manual for the selection of the best combinations of glaze frits and glazes for products of fine and structural ceramics.

## II. INDUSTRIAL DEVELOPMENT OF GLAZE PRODUCTION

Modern glazes are mixtures of complex silicates and borates. They are compounded in order to produce a coating on ceramic products and shall possess among other things:

- 1/ insolubility in water and usual acids and alkalis likely to be met in use;
- 2/ resistance to scratching;
- 3/ impermeability;
- 4/ resistance to crazing, peeling and similar faults;
- 5/ suitability for producing certain decorative effects, such as colour, crystal development, etc.;
- 6/ fusibility within predetermined temperature ranges.

One of the basic viewpoints for glaze classification is the necessity of fritting components soluble in water and their conversion to insoluble glass by melting with the other components. Such glazes are called fritted glazes. They are compounded mostly from a frit and an addition of plastic washed kaolin directly into a ball mill where they are ground to a required fineness. On the contrary, raw glazes do not contain components soluble in water and are prepared by grinding raw materials without preceding thermal treatment.

The advantages of the fulfritted glazes consist in the following experience:

- 1/ Raw materials reacting slowly with the other components at lower temperatures exhibit a higher melting effect after fritting.
- 2/ Fritting increases the temperature interval in fritting glazed products.
- 3/ Fritting decreases the firing temperature of glazes. It enables the application of glazes with a higher content



of oxides  $Al_2O_3$  and  $SiO_2$  which increase the resistance to abrasion and often to crazing.

- 4/ Fritting enables the lead oxides in glaze composition, which can be a source of dangerous poisonings, to be reduced to a minimum or eliminated completely.
- 5/ Fritting enables the application of selected raw materials soluble in water.
- 6/ Underglaze decorations are more distinct and give greater contrast under fritted glazes with regard to their lower chemical reactivity.
- 7/ Fritted glazes simplify storing of raw materials, charging into the mill and reduce the dispersion of properties of particular charges.
- 8/ Fritted glazes melted at lower firing temperatures give products with finer and brilliant surface.

A further evident development in the production of glazes is the diversion from application of lead glazes induced not only by high prices of lead containing raw materials, but also by the possibility of poisoning by lead. These factors were applied in spite of the fact that lead glazes have some technological advantages if compared with leadless glazes.

- 1/ Leadless glazes are fired within narrower firing interval.
- 2/ The achievement of the optimum relation between glaze and body is usually more difficult with leadless glazes than with those containing lead. It is due to a higher content of alkaline oxides in leadless glazes; the increased content of these oxides is necessary for reaching required firing temperature.
- 3/ Lead glazes exhibit a higher degree of elasticity which can compensate the stress between glaze and body without appearance of hair lines.
- 4/ Leadless glazes are more inclined to recrystallization if fired in outdated types of kilns with a long cooling cycle.

5/ Leadless glazes show always a higher viscosity in liquid state when the degasification of bubbles is always more difficult than with lead glazes.

The elimination of lead glazes was also supported by updating and quick development of kilns where firing conditions are so defined that a series of technological disadvantages of leadless glazes is practically negligible.

The basic turn in the development of glazes was the transition from transparent to opaque glazes which enabled the development of fine and structural ceramics based on less valuable raw materials at higher surface whiteness of glazed products. While outside Europe most countries were oriented and some of them still stick to the principle of adding fine-ground opacifying agents direct into the mill, Czechoslovak plants introduced in the fifties the production of frits and glazes with fritted zirconsilicate which replaced deficient tin dioxide. Fritted zirconsilicate brings a series of technological advantages for the ceramic technology proper attaining higher whiteness, lustre and brilliancy of surface.

One of the further characteristic features in the development of glazes is the reduction of firing temperature which is not only a contribution from the energy point of view but which enables in a series of cases the development of a composition giving a more perfect surface. However, most of these low temperature firing glazes are derived by addition of alkaline oxides that raise the coefficient of thermal dilatation of glaze and hence require the change of rational body composition.

A glaze may be satisfactory for a given temperature in one plant, but may be unsatisfactory at the same temperature in another plant because of the following variables:

- 1/ Firing cycle
- 2/ Body composition and type of product
- 3/ Physical properties and chemical formula of raw materials
- 4/ Kiln atmosphere, type of fuel used, setting, etc.
- 5/ Technique of application and glaze preparation

Although laboratory trials usually give a good indication of the suitability of a glaze for a specific application, it is advisable to run plant trials before deciding on the final glaze composition.

### III. RAW MATERIALS AND OTHER COMPONENTS

The raw materials of glazes may be broadly divided on the basis of so-called molecular formula into:

- a/ materials supplying the basic oxides which will flux with  $\text{SiO}_2$  and  $\text{B}_2\text{O}_3$  to form glassy materials containing complex silicates and borates;
- b/ materials supplying  $\text{Al}_2\text{O}_3$  (amphoteric oxides);
- c/ materials supplying  $\text{B}_2\text{O}_3$  and  $\text{SiO}_2$ .

In addition, there are special additions of such opacifiers, crystallizing agents and colouring agents.

The following glaze constituents are used for glaze production:

#### Silica ( $\text{SiO}_2$ )

Source: Feldspar, flint and clay

Silica may be adjusted to regulate the melting temperature of the glaze. Varying the RO group and balancing the group against any desired silica content, the maturing temperature of the glaze may be closely controlled. By increasing silica at the expense of boric acid, a glaze is made harder, more brilliant and more durable. It is said that substituting silica for boric acid will increase the coefficient of expansion; consequently, relative amounts of boric acid and silica may be proportioned to achieve the best possible "fit" between body and glaze.

Boric oxide (B<sub>2</sub>O<sub>3</sub>)

Source: Borax and boric acid

This is an important constituent of glazes because it produces low melting mixtures while at the same time forming almost as good a glass as silica. When it is used in large percentages, it lowers resistance of the glaze to acid and water attack. It is a good flux, reduces the viscosity within limits, and promotes high gloss. As stated above, boric oxide and silica may be varied to achieve better glaze "fit". High boric oxide reduces glaze hardness, increases the glaze solubility, shortens the firing range and causes running of colours. Glazes containing B<sub>2</sub>O<sub>3</sub> are usually not suitable for long, direct-firing conditions such as are encountered in heavier clay products (brick, tile, etc.).

Alumina (Al<sub>2</sub>O<sub>3</sub>)

Source: Feldspar, cryolite or alumina hydrate, kaolins and clays

Alumina can combine with silica or the basic oxides and is widely used as a means to regulate the oxygen ratio, thereby preventing devitrification. In bright, glossy glazes the alumina to silica ratio usually varies from 1:6 to 1:10, while in matte glazes the ratio ranges from 1:3 to 1:4. Increasing the alumina is usually considered to be the best method of obtaining good mattes. In the glossy type of glazes, the alumina content regulates the degree of fluidity of the molten mass (an increase of alumina increases the viscosity), and gives it the stability needed at the temperature required for maturity. It has a greater effect on durability than any other single oxide, increases refractoriness, improves strength, lowers the thermal expansion and adds hardness and brilliance to the glaze.

Soda (Na<sub>2</sub>O)

Source: Feldspar, Borax, sodium carbonate and sodium nitrate

Soda, an alkali, is a somewhat more powerful flux than potash and is used mostly in very low-temperature glazes where the effect of its fluxing power is required. Sodium glazes are usually less resistant to water than the corresponding potash glazes. Sodium has a high coefficient of expansion and may cause crazing. It decreases the tensile strength and the elasticity as compared with the action of the other bases commonly used.

Lime (CaO)

Source: Pure calcium carbonate, burned lime, whiting, limestone, dolomite and anorthite (lime feldspar)

Lime is the principal flux in medium- and high-temperature glazes, however, it is not as effective at lower temperatures. (Below Cone 4). It reduced the viscosity in glazes having a high silica content, however, if liquid is too fluid it will have a tendency to devitrify easily, giving a dull matte finish. It lowers the coefficient of thermal expansion as compared to the alkalis. It promotes adherence of glaze to the body, since it reacts with both body and glaze. Calcium contributes stability, hardness and toughness to the glaze when used in proper amounts.

Lead (PbO)

Source: Red lead (Pb<sub>3</sub>O<sub>4</sub>), white lead (2 PbCO<sub>3</sub>.Pb(OH)<sub>2</sub>) and litharge (PbO)

Lead reacts easily with silica to form low-melting lead silicates which form stable glasses having high lustre and brilliance due to a high refractive index. It has a low

coefficient of expansion as compared to the alkalies, decreases the viscosity of the melt, and makes the glaze less likely to devitrify. Lead glazes are easy to use and are not as "tender" as other types; that is, small irregularities in processing may not harm a lead glaze where they would make worthless a ware coated with a leadless glaze. When used in the glaze as a powdered frit, the silicate produces a strong fluxing action. It is relatively insoluble in water. This is particularly true of firing, since lead glazes have a longer firing range. The use of lead compounds in glazes also has some disadvantages, such as:

- 1/ Poisonous nature of many of its forms.
- 2/ Improperly constituted glazes can be susceptible to attack and solution by fruit juices and other foodstuffs.
- 3/ Volatilizing easily during firing, thus promoting loss of gloss.
- 4/ Crazeing common but usually avoidable.
- 5/ Dimming of the brilliance after long exposure to gases commonly present in the atmosphere.
- 6/ Abrasion resistance decreases as the content of lead rises.
- 7/ Subject to reduction in a reducing kiln atmosphere.

#### Potash ( $K_2O$ )

Source: Most common source is feldspar, since silica and alumina are also introduced. Other sources are potassium carbonate and potassium nitrate.

Potash, an alkali, is a very common flux often preferred to soda because it produces a more brilliant glaze and gives a slightly longer firing range. It increases glaze expansion and will cause crazing if used in large amounts.

### Barium (BaO)

Source: Barium carbonate

Barium is mostly used in lead-free formulae. It is also used as a flux in the formation of a matte structure. In larger amounts, it takes the role of a refractory, increasing the melting temperature. In extremely small amounts it improves the gloss and mechanical strength and aids resistance to organic acids to some extent.

### Magnesium (MgO)

Source: Magnesium carbonate, dolomite and talc.

Magnesium acts as a refractory up to a relatively high temperature, however, a mixture of lime and magnesium as in dolomite is not as refractory as magnesium alone. At high temperatures it becomes a very active flux which increases fluidity of the glaze and lowers thermal expansion. It behaves similarly to lime, making a glaze more fluid at high temperatures. It is used as a constituent of some low-temperature mattes, it tends to reduce crazing and may be harmful to certain underglaze colours. Although magnesium is not a strong enough flux to replace lead, it is valuable in a leadless glaze in small amounts.

### Lithium (Li<sub>2</sub>O)

Source: Lithium carbonate, lepidolite, ambligonite and spodumene

Lithium, an alkali, is a powerful flux and is most valuable in leadless glaze compositions. Its high cost and thermal expansion prohibits the use of large percentages of lithium in a given composition, however, small amounts will produce marked changes in the fusibility, fluidity and surface tension of a glaze.



### Strontium (SrO)

Source: Strontium carbonate

As is the case with barium, strontium is used to a limited extent in the same manner as lime and magnesium. It appears to offer all the advantages of barium in glazes without the poisonous properties, and has the tendency of barium to cause defects in a kiln atmosphere containing sulphur. It may be used as a replacement for lead in low-temperature glazes, but a longer heat soak is necessary during glaze firing. Strontium extends the firing range of certain high lime glazes and should improve glaze "fit", hardness and decrease solubility expansion when substituted for either calcium, zinc or lead on a molecular basis.

### Zinc (ZnO)

Source: Zinc oxide in pure form

Zinc is used in many glazes as a flux to prevent crazing. In moderate amounts zinc often improves the elasticity (toughness), lengthens the maturing range and enhances a brilliance of colours, such as blues, chrome alumina pinks and browns. It will produce opacity or whiteness, providing the calcium content is low. As the zinc content of the glazes increases, matteness is developed. Zinc lightens blues, warms browns and injures copper and chrome greens and chrome-tin pinks. Zinc may harm underglaze colours.

### Kaolins and clays

Clays are indispensable as glaze materials because:

- 1/ They assist in keeping other glaze materials in suspension in the slip.
- 2/ They are largely responsible for causing adhesion of the unfired slip to the ware.

3/ They are a medium for control of the shrinkage of the unfired slip upon the ware.

For transparent glazes, clays must contain a minimum amount of impurities and consequently china clays and kaolins are preferred. As a matter of interest, the setting properties of clays may be grouped as follows:

Florida kaolin:	Relatively low set
Ball clay:	Medium set
Bentonite:	High set

Often by the proper choice of clays the use of an electrolyte can be minimized and certain bisque defects such as cracking, peeling and dusting may be avoided.

#### Feldspars

Unlike clays, the theoretical formula of feldspar should not be used in the calculation from empirical to batch formulae. All feldspars are varied combinations of soda spar (albite) and potash spar (orthoclase or microcline). The analyses of various spars can be secured from the supply houses. The physical properties of the glaze may be altered by simply changing from one spar to another. The substitution of potash spar for soda spar will decrease the fluidity and coefficient of expansion but it will also increase the resistance to solution by water and increase scratch resistance. Feldspar acts either as a flux or as a refractory, depending upon the other batch constituents and the maturing temperature of the glaze.

### Setting-up agents

The function of setting-up agents is threefold:

- 1/ To prevent glazes from settling into a hard deposit at the bottom of the container.
- 2/ To prevent glazes from becoming too fluid or runny and from dripping off the pieces after dipping, especially on vitreous ware.
- 3/ To prevent curtaining - the uneven distribution of the glaze layer.

Some of the common agents are:

- 1/ Clay - In the finer products this amount should be kept at a minimum, just enough to keep the glaze in suspension.
- 2/ Acetic acid - Acetic acid has been used successfully in some cases; however, care must be taken, since an excess will thin the glaze out beyond the range of good dipping practice.
- 3/ Magnesium sulphate - Although this is the most popular electrolyte, it must be remembered that sulphates are introduced into the glaze. It should be added into the glaze in solution and not in crystal form.
- 4/ Calcium chloride - Like magnesium sulphate, it is very effective.
5. Bentonite - May be used at approximately 1% in all fritted glazes where clay and electrolyte are kept at a minimum.
6. Barium chloride

### Gums

Dextrine, gum arabic, gum tragacanth, gum ghatti and gum karaya are some of the more common organic substances which are useful in promoting adhesion and preventing dusting of the glaze in the dry state. A solution of gum and water should be made beforehand and added to the glaze slip, since direct introduction of the gum will form separations of sticky masses.

Methyl cellulose is used as a glaze slip ingredient in dinnerware, sanitary ware and tile in both dip and spray applications. It is a film former, hardener and binder as well as a suspending agent and leveler. It reduces glaze rejects by facilitating handling, diminishing running and sagging and improving binding and producing a more uniform finish.

#### Opacifiers

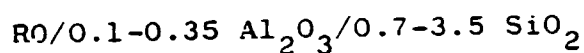
The effect of chrome flashing that is noted in tin oxide opacified glazes is not present in glazes containing zirconium type opacifiers. When these opacifiers are used with chrome based stains and colours, no major chrome change is produced in the full range of tones from light pastels to the full stain intensity.

The opacification of glazes may be accomplished by means of a number of different zirconium products. The synthetic double silicates opacifiers produce good opacity singly or in combination. However, where they are used in combination, there is an added advantage of better slip properties producing easier application. Zirconia opacifiers ( $ZrO_2$ ) have been found beneficial in promoting opacity when used in combination with zircon opacifiers ( $ZrSiO_4$ ), by affording a means of saturating the glaze.

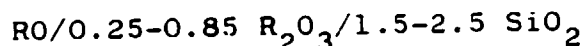
#### IV. GLAZE COMPOSITIONS

Examples of general types of glaze ranges:

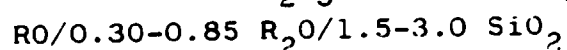
a/ Raw lead glaze



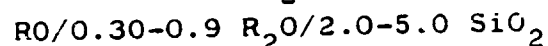
b/ Fritted glazes



Cone 010-06

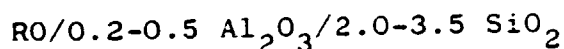


Cone 05-01

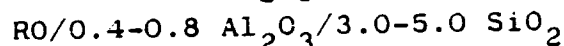


Cone 1-6

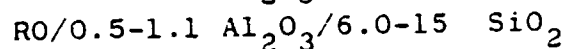
c/ Raw leadless glaze



Cone 1-6

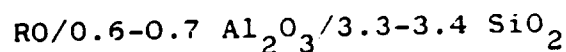


Cone 7-10



Cone 11-16

d/ Salt glaze



Cone 4-9

These ranges are very general and serve mainly to show the ratio of RO oxides to alumina and silica and the ratio of alumina to silica. Actually, the greatest difference lies in the RO group consisting of RO and R<sub>2</sub>O oxides. The properties of the glaze may be made to vary over a wide range by the choice of oxides from this group. For example, in the lowest firing glazes the RO group usually consists of soda, potash, lead, calcium, magnesium and zinc. The glaze may not contain all of these oxides but usually contains at least three or four of them with lead and potash predominating in the lower melting compositions.

As the fusion point is raised, the refractory oxides predominate and combinations such as potash and soda may be replaced to a certain extent by a single oxide. Boric oxide is commonly used to replace part of the silica. In the medium firing range, the principal flux is feldspar aided by lead and calcium. In porcelain glazes, feldspar is the main flux. Only in the lower fusing compositions of porcelain glazes is any auxiliary flux used.

The principle of salt glazes is based on the effect of alkalis in gaseous state on the surface layer of ceramic products. A thin layer of glaze develops during ware firing consisting of alumino-alkalic silicates, which covers the whole surface of product. The glazing technique with salt glazes is quite different from the application of all the other glazes. It consists in throwing relatively small quantities of normal salt into the hearth of a chamber kiln or in dispersing it into the firing zone of a tunnel kiln. The vapours of salt decompose by the action of water vapours contained in gases and HCl and Na<sub>2</sub>O are created. The vapours of hydrochloric acid are more volatile and escape with the other gases from kiln while the vapours of sodium oxide react with a part of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> from the body which creates the glaze coating. A good salt glaze requires a high SiO<sub>2</sub> content in the body reaching with Al<sub>2</sub>O<sub>3</sub> the ratio 1:4 to 1:10. The lower is this ratio, the higher must be the firing temperature of stoneware at which the salt of good quality is created.

## V. COLOURED GLAZES

The glazes are coloured in principle by three methods:

- 1/ By fritting of colouring elements into the frit which enables on one side a perfect homogenous colouring of glaze and decreases on the other side thermal decomposition and volatilization at higher temperatures. This technology is applied with some cobalt and practically with all selenium-cadmium glazes.
- 2/ By the preparation of stains, i.e. substances with high content of colouring agents; these are added directly to a frit in a mill. This technology is the most usual in ceramic practice and enables to win a series of shades of glazes by different additions of stains or by combination of different stains.

Stains are coloured silicates the coloured components of which ( $\text{Na}_2\text{O}$ ,  $\text{K}_2\text{O}$ ,  $\text{CaO}$ ,  $\text{SrO}$ ,  $\text{BaO}$  and  $\text{ZnO}$ ) are partly or completely replaced by colouring oxides.

The production of stains is an independent complicated branch of physical chemistry. Below, examples of colouring agent combinations with carriers and resulting colours are listed.

In the modern practice, the influence of so-called trace elements is applied which if added to stains, increase their colouring effect.

Main components of some ceramic stains:

<u>Basic colour</u>	<u>Colouring oxides</u>	<u>Carriers</u>	<u>Additions</u>
blue	cobalt, vanad	$Al_2O_3$ , $SiO_2$ , $ZnO$ , $ZrO_2$	fluorides
green	vanad, cobalt, chrome	$ZnO$ , $Al_2O_3$ , $SiO_2$	
brown	manganese, chrome, iron	$ZnO$ , $Al_2O_3$ , $SiO_2$ , alum	
red	chrome, manganese	$SiO_2$ , $Al_2O_3$ , $Al(OH)_3$ , $H_3PO_4$ $ZnO$ , $SnO_2$ , $CaCO_3$	fluorides
grey	antimony, carbon	$SnO_2$ , $Al_2O_3$ , $SiO_2$	oxalic acid
yellow	uranium, vanad, antimony	$SnO_2$ , $Al_2O_3$ , $ZrO_2$ , $SiO_2$ , $Pb_3O_4$	Ca compounds

Stains are finely ground, usually to 3 - 5 micrometers to be more effective. Most colouring oxides act in dissolution in glazes as a strong flux, they participate in the creation of glass and can change in higher additions the properties of glaze in such a way that a correction of glaze is necessary, usually by  $SiO_2$  addition.



3/ By the application of thermally stabilized colouring metal oxides as e.g.  $\text{MnO}_2$ ,  $\text{CuO}$  etc. However, this technology in the stabilized ceramic production requires pre-grinding of colouring oxides with a small quantity of frit or quartz. The obtained concentrate is then added to a frit.

A required shade of glaze colour must always be tried experimentally as the glaze composition influences the shade of the achieved colour. Basic differences are achieved by the application of the same addition of one stain to a transparent or opaque glaze. The combination of white opacification with a coloured one is used for the achievement of pastel colours for opaque glazes. Deep and expressive hues of colours for transparent glazes are achieved especially if the glaze contains lead oxides. Undesirable shades occur often in the production of coloured glazed ceramic ware fired in large tunnel kilns. These shortcomings depend on:

- a/ firing temperature
- b/ kiln atmosphere
- c/ glaze thickness
- d/ regularity of glaze thickness
- e/ frit composition
- f/ fast applied stains
- g/ equal fineness of stain and glaze

The lowest quantity of shades is achieved in one-layer fast firing of glazed ware.

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