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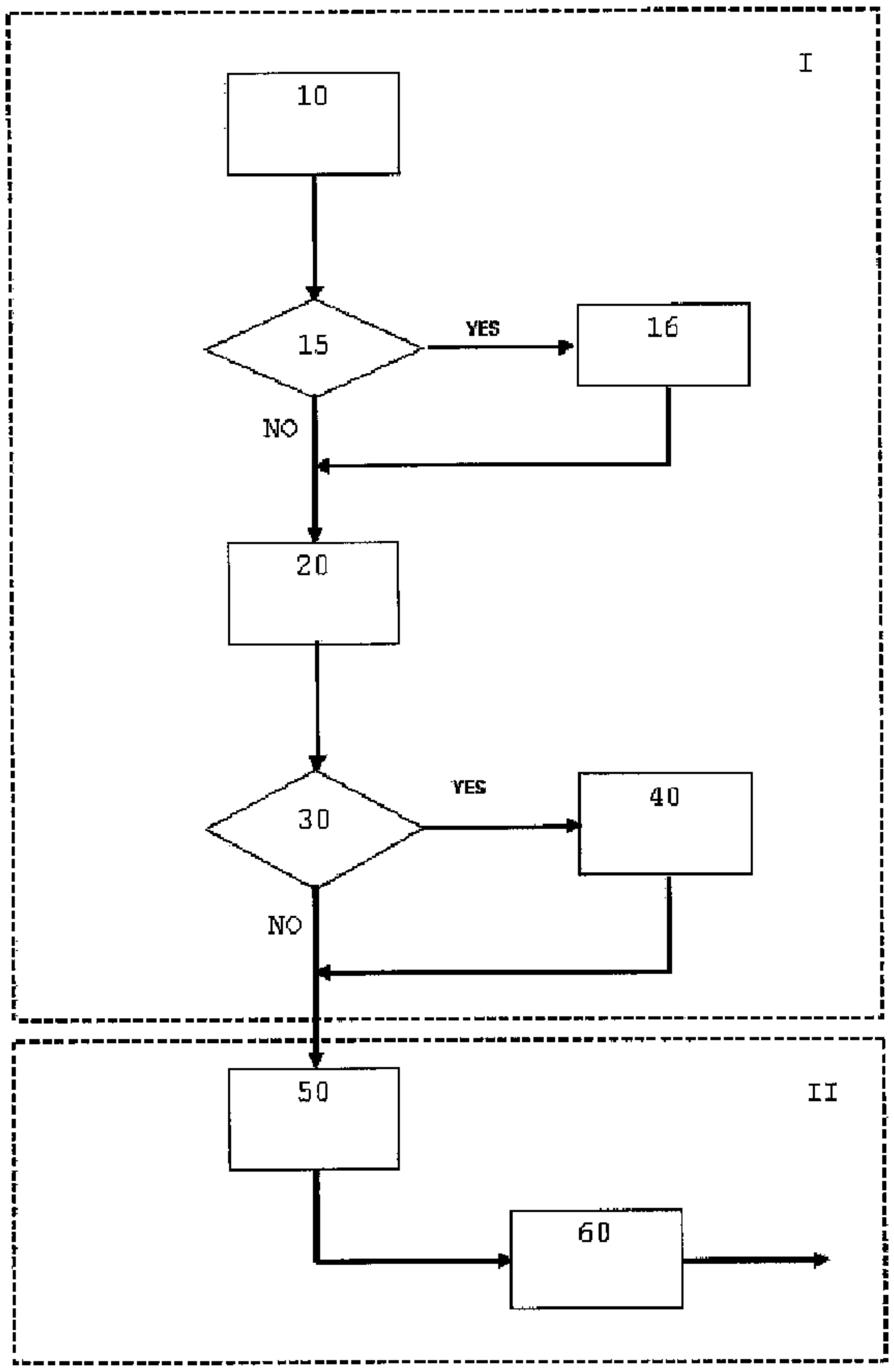
(54) **Titre** : PROCEDE DE PREPARATION D'ADDITIF IGNIFUGEANT DESTINE A DES REVETEMENTS ET PRODUITS AINSI OBTENUS
(54) **Title**: METHOD FOR PREPARING A FIRE RETARDANT ADDITIVE FOR COATINGS AND RESULTING PRODUCTS



(57) **Abrégé/Abstract:**

The invention relates to a method for preparing an additive based on metal hydroxide and, in particular, magnesium hydroxide, to be integrated into coatings in order to provide same with improved fire retardant properties. The average particle sizes of the

(57) Abrégé(suite)/Abstract(continued):

hydroxide are selected from between 1 nanometre and 10 micrometres, preferably with a very small variation. The magnesium hydroxide is subjected to a washing and dispersion treatment so that it disperses efficiently and does not interfere with the desirable properties of the coating. The materials and conditions used in the treatment and the particle size are selected according to the type of coating. The end coating can be based on water, solvents, oil and alcohol. Compared to coatings formulated with other fire retardant compounds, smaller weight losses due to burning have been demonstrated with the inventive additive in an ASTM D1360 standard test.

ABSTRACT OF THE INVENTION

The invention relates to a method for preparing an additive based on metal hydroxide and, in particular, magnesium hydroxide, to be integrated into coatings in order to provide same with improved fire retardant properties. The average particle sizes of the hydroxide are selected from between 1 nanometre and 10 micrometres, preferably with a very small variation. The magnesium hydroxide is subjected to a washing and dispersion treatment so that it disperses efficiently and does not interfere with the desirable properties of the coating. The materials and conditions used in the treatment and the particle size are selected according to the type of coating. The end coating can be based on water, solvents, oil and alcohol. Compared to coatings formulated with other fire retardant compounds, smaller weight losses due to burning have been demonstrated with the inventive additive in an ASTM D1360 standard test.

METHOD FOR PREPARING A FIRE RETARDANT ADDITIVE FOR COATINGS AND RESULTING PRODUCTS

TECHNICAL FIELD FOR THE INVENTION

The present invention is related to the procedures for the preparation of additives for use in coatings applied to structural elements or as finishes for materials susceptible to combustion, such as wood and its derivatives, with the purpose being to provide properties that are flame retarding once the material to be protected is covered; specifically, the invention refers to a procedure for the preparation of an additive formulated with a metallic hydroxide and a mix of a dispersant, a compatible agent (an organize solvent), water and resin that allow the easy incorporation of the additive into the coating.

PRECEDENTS TO THE INVENTION

The need for residential buildings to be constructed or for the realization of other activities: work, recreation, etc. incorporating means that help control fires and slow the expansion of the fire is well known. For example, some countries have released regulations on materials for the construction of buildings, as much as for the fabrication of the furniture that is contained within them.

The majority of the furniture has coatings that have esthetic purposes as well as for the protection the material which it is made from, nonetheless, the majority of these coatings are inflammable, which when added to the natural combustible property of the base material, generates an excellent source for the spreading of the fire.

Coatings that are flame retarding can be found, they basically are made of phosphates, silicates, borates (intumescent). An expanding flame retarding material such as those mentioned is characterized because with the action of fire, first, it forms a foam layer that transforms into ash that then forms a crust that impedes the penetration of the fire into the base material.

The metallic hydroxides, on the other hand, are used as flame retarding additives, for example, in polymers, with various different applications: coatings for cables, furniture, roofing tiles "roofing", etc. The magnesium hydroxide has the advantage over other types of flame retarders because it is environmentally friendly and it does not release toxic fumes when utilized as a smoke suppressant.

There already exists a large variety of alternatives to control the problem of fire expansion, through the use of metallic hydroxides. Some examples are mentioned below.

In patent EP 1156092, it describes the fabrication of an adhesive of two components that is created by using magnesium hydroxide, mentioning that said adhesive can be used as a varnish with flame retarding properties.

In the United States patent 6448308, it includes magnesium hydroxide as part of the formulation of a coating that is flame retarding that has a base of red phosphorus. The use of the magnesium hydroxide is specifically used for reducing the production of phosphoric acid during the process of inhibiting the flames.

In United States patent number 4849298, it describes the use of aluminum hydroxide in the creation of paints. It is added directly to the paint and also, it is used as a substitute in some of the conventional types of paint (TiO2, for example). The patent describes the effect of the flame retarding properties of coatings prepared with this additive.

Upon adding the proper amount of previously treated magnesium hydroxide to the traditional coatings for wood, such as varnishes and sealers, or beautifiers, such as paints, water proof coatings, etc. it achieves excellent results as a flame retarder and a smoke suppresser. The coating thus prepared does not change its final appearance (shine, transparency, covering power, mechanical resistance, etc.)

Objectives of the Invention.

In light of the problems seen in the previous inventions, one objective of the present invention is to provide an additive that is a flame retarder and a smoke suppresser when used in the preparation of coatings designed to cover and protect surfaces that are susceptible to combustion, and whose efficiency would be better than those offered by the present flame retardant coatings.

Another objective of the present invention is to provide a flame retarding agent that is easily incorporated into the coating before being applied to the surface it is going to protect.

One more objective of the present invention is that the additive incorporates as the flame resistant ingredient a material that does not generate toxic residues when such is the case that it is exposed to flames.

Another objective of the present invention is that the flame retarding ingredient be of the family of metallic hydroxides.

Still another objective of the present invention is that the flame retarding ingredient of the additive of the invention does not form groupings in the interior volume of the coating before its application.

One more objective of the present invention is the flame retarding ingredient of the invention´s additive be distributed in a uniform (homogeneous) manner in the film of the final coating on the surface to be covered and protected.

Another objective of the present invention is that the flame retarding additive does not interfere with the fundamental properties of the coating to which it is added, as an example, the transparency of a varnish.

One more objective of the present invention is the flame retarding ingredient of the additive presents a grain similar to that of the coating in which it is included.

A SHORT DESCRIPTION OF THE INVENTION

The additive of the coating to the prepared with the procedural ingredient of the invention has as a source of its flame retarding properties a metallic hydroxide, preferably of magnesium, with individual characteristics according to the size of the particle, that is submitted to a treatment associated with the nature of the coating into which it will be incorporated, in a way that the resulting additive is able to be efficiency integrated into the coating selected.

Some characteristics of the additive created with the procedural ingredient of the invention are:

a) An average diameter (D_{50}) of the particle of the metallic hydroxide is selected from a range of from between 1 nanometer to 10 microns, depending on the function of the finish desired of the coating
b) The concentration of said particles of metallic hydroxide in the additive can have up to 99% of the weight.

The invention´s procedure produces a paste or preferably a suspension of particles of metallic hydroxides treated with other compatible agents (organic solvents or water), dispersants and eventually surfactants, in a substance made up of an organic solvent, a dispersant, a resin and in some cases water, that show the following advantages:

1. To offer better efficiency in the flame retardance of the intumescent additives, that in previous comparative tests of the flame retardance show to obtain better results in the coatings mainly with magnesium hydroxide than those with a base that is composed of phosphates. The results are expressed as a percentage of loss of weight per action of the flame.
2. To be easy to incorporate into the coatings that already exist on the market, without needing to use equipment different from that which is conventionally used for the preparation of coatings, such that the additive has a base that is compatible with the coating into which it will be incorporated.
3. To not alter the final appearance of the coating, which is different from the flame retardants that have a base of phosphorus, in which it is necessary to apply an additional coating that provides the final appearance desired from the finish. With the additive of the invention the characteristics of the shine, the transparency, the ability to cover, the mechanical resistance, etc., of the coating will not be altered.
4. To be environmentally friendly and not harmful to living creatures. In various studies it has been demonstrated that the use of metallic hydroxides in flame retardant coatings have advantages over products such as those that are used as halogenous and phosphoric compounds, and that do not give off toxic gasses, and the release of water in the reaction of the decomposition of the hydroxide helps control the smoke emissions.

5. The surface treatment given to the magnesium hydroxide is selected from a series of surface additives that help make the magnesium hydroxide compatible with the resin of the additive and the coating object, maintaining the characteristics, such as the shine, transparency, covering power, mechanical resistance, etc., of the coating object.

DESCRIPTION OF THE FIGURE

For a better understanding of the description that follows, the attached figure should be regarded when reading the description.

Figure number 1 is a diagram of blocks that represent the procedure for the creation of the additive, the purpose of the invention.

DETAILED DESCRIPTION OF THE INVENTION

The additive prepared according to the procedure of the present invention is created from a metallic hydroxide with an average particle size selected from the range of 1 nanometer to 10 microns, preferably being monodisperse, and having a wide variety of sizes and with a purity of at least 90%.

The metallic hydroxide particles are submitted in a treatment for their integration into the final coating, for that which can be separated from a hydroxide in a diluted suspension, being organic or in dust, without that the compatibility between the initial hydroxide agent and the base of the additive that will formed be limiting to it.

In the preferred form of the present invention, the metallic hydroxide is magnesium hydroxide. It has as an advantage in addition to its flame retarding properties, that it helps control the emission of fumes upon releasing the water when the elements in the reaction break down (1);

$$Mg(OH)_2 \xrightarrow{\Delta} MgO + H_2O \qquad (1)$$

With reference to figure 1, which is a diagram of blocks of the process of the production of the additive of the invention, it is presented in two zones: the first composed of the blocks from (10) to (40) that represent the pretreatment of the particles of metallic hydroxide, and the zone composed of blocks (50) and (60) representing the process of the preparation of the additive itself.

In zone I, or the pretreatment phase, the block (10) represents the flame retarding ingredient that will be used for the preparation of the additive, preferably being a wet paste of metallic hydroxide although for some very specific applications that require the absence of water, it is preferable to use a hydroxide in a dry powdery form. As was mentioned before, the hydroxide is formed by particles with an average size in the range from 1 nanometer to 10 microns and with a purity of at least 90%. This material is added to the following block.

The block (20) represents an operation called the "change of agent" in which the wet paste of the metallic hydroxide is washed with the purpose of displacing the water contained and substituting it for a "compatible" organic solvent, that is, it is incorporated without provoking separation of the stages, with the solvent or the thinner of the final application (the "objective" coating), which will stop in the moment the formation of lumps when it comes in contact with the objective coating; the process takes place with strong agitation, preferably for between 5 and 30 minutes, or for the time deemed necessary. The mix will be maintained in agitation in a turbulent pattern by means of a diffuser with a sharp disk or other device that allows the peripheral velocity to be at least 2 m/sec and up to 30 m/ sec. After the agitation, the separation of the stages results and the process can be repeated until a residual humidity of less than 5% is obtained when in the solid state.

When by the nature of the solvent or the thinner and of the resin of the final application, the metallic hydroxide could react, the need is evaluated, as is indicated by the block (15), in order to give the particles a surface treatment (16) before the operation of the "change of agent" (20), using similar conventional surfactants with the objective coating.

The process of the "change of agent" (20) serves to guarantee that the hydroxide particles do not group together in the diffusion phase (50) in Zone II, in the incorporation to the coating or in the application of the same on to the surface to be treated.

The block of decision (30) indicated that in case that the tolerated residual humidity in the additive is very low, close to zero, due to the nature of the resin and solvents or thinners in the objective coating and once the "change of agent" (20) stage is complete, the solid phase is summated to a process of drying (40), where the temperature of the operation in said drying stage always needs to be lower than the boiling point of the agent. The operation continues until the tolerated residual humidity for the objective coating is obtained.

The result of the operation is a "dry" power of hydroxide that can be stored for the later preparation of the additive. The product obtained by this means can remain as a "dry" power dispersible for periods of 12 months.

If in the previous step the humidity content of 5% is reached, the drying stage that is represented by block (30) is omitted.

The product obtained, be it "dry" or wet, coming from one of the two previous steps of the first phase of the process then passes to a process of diffusion (50), in Zone II, that is adequately identified with the preparation of the additive ready for use in the objective coating that is the material of the present invention.

In this stage, the paste or the "dry" powder coming from blocks (20) or (30), are added to a process of diffusion (50) where a resin and a similar dispersant is added to the objective coating, according to the following table:

TABLE 1. Recommended selection of the resin and the dispersant for the preparation of the additive, taking into account the nature of the coating

TYPE OF OBJECTIVE COATING	RESIN RECOMMENDED FOR THE DIFFUSION	DISPERSANT RECOMMENDED FOR THE DIFFUSION
Polyurethane	Polyester	Copolymer with acidic groups, ethyl ammonium chloride salt of a polycarboxylic acid, ethyl ammonium chloride salt from a non saturated fatty acid, non saturated polyamine amide salt and polyester acids with low molecular weight, unsaturated polyamine amide and polyester acids of low molecular weight
UV Curing	Epoxy-acryate	Copolymer with acidic groups, ethyl ammonium chloride salt of a polycarboxylic acid, ethyl ammonium chloride salt of a non saturated fatty acid, non saturated polyamine amide salt and polyester acids with low molecular weight, unsaturated salt of polyamine amide and polyester acids with low molecular weight
Acrylic-styrene	Acrylic- styrene	Ammonium salts of an acrylic copolymer, salt of ethyl ammonium chloride and a multifunctional polymer of a anionic character, sodium salt of an acrylic copolymer
Vinyl	Vinyl	Ammonium salt of an acrylic copolymer, salt of ethyl ammonium chloride and a multifunction polymer with an anionic character, sodium salt of an acrylic copolymer

TYPE OF OBJECTIVE COATING	RESIN RECOMMENDED FOR THE DIFFUSION	DISPERSANT RECOMMENDED FOR THE DIFFUSION
Ammonium chloride Enamel	Ammonium chloride Resin	Copolymer with groups of acids, ethyl ammonium chloride of a polycarboxiclic acids, ethyl ammonium chloride of a non saturated fatty acid, non saturated polyamine amide salt and polyester acids with low molecular weight, unsaturated polyamine amide and polyester acids of low molecular weight
0% composed of organic volatiles	Acrylic- styrene. Vinyl, Vinilica, Acrylic epoxy	Ammonium salt of an acrylic copolymer, salt of ethyl ammonium chloride a multifunctional polymer with an anionic character, sodium salt of an acrylic copolymer
Nitrocellulosic	Ethyl ammonium chloride or Stabilized nitrocellulose	Copolymer with acidic groups, ethyl ammonium chloride salt of a polycarboxylic acid, ethyl ammonium chloride salt from a non saturated fatty acid, non saturated polyamine amide salt and polyester acids with low molecular weight, unsaturated polyamine amide salts and polyester acids of low molecular weight
Alkaline	Alkaline of soy or coconut, lecithin	Copolymer with acidic groups, ethyl ammonium chloride salt of a polycarboxylic acid, ethyl ammonium chloride salt from a non saturated fatty acid, non saturated polyamine amide salt and polyester acids

TYPE OF OBJECTIVE COATING	RESIN RECOMMENDED FOR THE DIFFUSION	DISPERSANT RECOMMENDED FOR THE DIFFUSION
		with low molecular weight, unsaturated polyamine amide and polyester acids of low molecular weight
Phenolic	Phenolic Resin	Copolymer with acidic groups, ethyl ammonium chloride salt of a polycarboxylic acid, ethyl ammonium chloride salt from a non saturated fatty acid, non saturated polyamine amide salt and polyester acids with low molecular weight, unsaturated polyamine amide and polyester acids of low molecular weight

The diffusion (50) is done by means of an agitator or a disperser having a peripheral velocity of between 15 and 30 m/sec. The viscosity of the mix is adjusted to that of the objective coating for the addition of the solvent or the thinner that preferably is of the same base that will be used with the coating or at least should be compatible with it. The percentage of dispersant in the mix is maintained between 0.5 and the 10% in relation to the hydroxide in the dry base.

The product (60) obtained from the process of the diffusion (50) is the additive of the invention, the said product that can be in the preferred form, a formulation with up to 99% of its weight in metallic hydroxide.

Among the advantages that the additive has obtained by the process of the invention are that as a consequence of the treatment of the change of agent in stage (20) and the mix with resins and dispersants in stage (50), the product is completely compatible with the objective coating for that which was prepared during the selection of the appropriate surfactant, resin and dispersant according to the table previously included, furthermore, maintaining a high grade of homogeny in the dispersion of the metallic hydroxide particles in the formation. For that which when added to the objective coating, the additive will integrate quickly and easily and it is guaranteed that the particles maintain the homogeny throughout the whole volume and thus, in the layer of the coating after the application onto the surface it is to protect.

In the preferred means of the intention, useful in the case of transparent finishes, particles with an average size in the range of nanoparticles with a low variability (monodisperse) are used with those that the properties of the transparency of the varnish are not altered, that which is not guaranteed with particles of larger sizes or with a non homogenous sample or a sample of wide dispersion.

For that reason, it is suggested that in the preparation of an additive for transparent varnishes to use particles from the extreme low end of the average size range. In the same way, with opaque coatings it is acceptable to use particles of larger sizes, and in the texturized finishes, particles from the extreme high end of the range can be used.

The use of particles from the extreme high end of the range (10 microns) does not guarantee a homogenous distribution in the layer of the coating on the surface that it is to protect, and thus, the flame retardant efficiency is considerably diminished.

Table 2 shows a summary of the selections of the reactors and the parameter values recommended for a variety of classes of conventional coatings used for common applications, with those that is possible to create additives, following the procedure according to the present invention , that will offer the characteristics of being easy to incorporate into the objective coating, homogenous (uniform) distribution of the particles of the flame retarding and smoke repressing ingredient, and conservation of the desired properties of the coating to name a few.

Table 2. Materials and parameters recommended for various types of coatings.

Type of coating		**Size of the $Mg(OH)_2$ Particles**	**Humidity in the initial hydroxide**	**Agent for the process of the "change of Agent"**	**Humidity of the Hydroxide before the diffusion**	**Dispersant for the diffusion**	**Solvent or thinner for the diffusion**	**Quality**
Varnishes	Nitrocellulose	1 to 190 nm	Less than 65%	Aryl Ketenes, aryl alcohols, Glycol ester, glycol ether, etc.	Less than 5%	Copolymer with acidic groups, ethyl ammonium chloride salt of a polycarboxylic acid, ethyl ammonium chloride salt from a non saturated fatty acid, non saturated polyamine amide salt	Ketenes, esters, alcohols, glycol esters, glycol esters, etc.	Less than 60 μm

Type of coating		Size of the $Mg(OH)_2$ Particles	Humidity in the initial hydroxide	Agent for the process of the "change of Agent"	Humidity of the Hydroxide before the diffusion	Dispersant for the diffusion	Solvent or thinner for the diffusion	Quality
						and polyester acids with low molecular weight, unsaturated polyamine amide and polyester acids of low molecular weight		

Type of coating	**Size of the $Mg(OH)_2$ Particles**	**Humidity in the initial hydroxide**	**Agent for the process of the "change of Agent"**	**Humidity of the Hydroxide before the diffusion**	**Dispersant for the diffusion**	**Solvent or thinner for the diffusion**	**Quality**
Ethylammonium chloride	1 to 190 nm	Less than 65%	Aryl Ketenes, aryl alcohols, glycol ester, glycol ether, etc.	Less than 5%	Copolymer with acidic groups, ethyl ammonium chloride salt of a polycarboxylic acid, ethyl ammonium chloride salt from a non saturated fatty acid, non saturated polyamine amide salt and polyester acids with low molecular weight, unsaturated polyamine amide and polyester acids of low molecular weight	Hydro carbonate, ketenes, esters, alcohols, glycol ethers, glycol esters	Less than 20 µm

Type of coating		Size of the $Mg(OH)_2$ Particles	Humidity in the initial hydroxide	Agent for the process of the "change of Agent"	Humidity of the Hydroxide before the diffusion	Dispersant for the diffusion	Solvent or thinner for the diffusion	Quality
	Phenolic	1 to 190 nm	Less than 65%	Aryl Ketenes, aryl alcohols, Glycol ester, glycol ether, etc.	Less than 5%	Copolymer with acidic groups, ethyl ammonium chloride salt of a polycarboxylic acid, ethyl ammonium chloride salt from a non saturated fatty acid, non saturated polyamine amide salt and polyester acids with low molecular weight, unsaturated polyamine amide and polyester acids of low molecular weight	Hydro carbonate, ketenes, esters, alcohols, glycol ethers, glycol esters	Less than 20 µm

Type of coating		Size of the $Mg(OH)_2$ Particles	Humidity in the initial hydroxide	Agent for the process of the "change of Agent"	Humidity of the Hydroxide before the diffusion	Dispersant for the diffusion	Solvent or thinner for the diffusion	Quality
	Polyurethane	1 to 190 nm	less than 65%	Aryl Ketenes, aryl alcohols, Glycol ester, glycol ether, etc.	less than 5%	Copolymer with acidic groups, ethyl ammonium chloride salt of a polycarboxylic acid, ethyl ammonium chloride salt from a non saturated fatty acid, non saturated polyamine amide salt and polyester acids with low molecular weight, unsaturated polyamine amide and polyester acids of low molecular weight	Hydro carbonate, ketenes, esters, alcohols, glycol ethers, glycol esters	less than 20 μm
PAINTS	Vinyl	1 to 5 μm	less than 5%	Water	less than 5%	Ammonium salt of an acrylic copolymer, salt of ethyl ammonium chloride a multifunctional polymer with an anionic character,	Water	less than 50 μm

Type of coating		Size of the $Mg(OH)_2$ Particles	Humidity in the initial hydroxide	Agent for the process of the "change of Agent"	Humidity of the Hydroxide before the diffusion	Dispersant for the diffusion	Solvent or thinner for the diffusion	Quality
						sodium salt of an acrylic copolymer		
	Acrylic Vinyl	1 to 5 µm	less than 5%	Water	less than 5%	Ammonium salt of an acrylic copolymer, salt of ethyl ammonium chloride a multifunctional polymer with an anionic character, sodium salt of an acrylic copolymer	Water	less than 50 µm

Type of coating		Size of the $Mg(OH)_2$ Particles	Humidity in the initial hydroxide	Agent for the process of the "change of Agent"	Humidity of the Hydroxide before the diffusion	Dispersant for the diffusion	Solvent or thinner for the diffusion	Quality
	Oil	1 to 2 μm	less than 5%	Aryl Ketenes, aryl alcohols, Glycol ester, glycol ether, etc.	less than 5%	Copolymer with acidic groups, ethyl ammonium chloride salt of a polycarboxylic acid, ethyl ammonium chloride salt from a non saturated fatty acid, non saturated polyamine amide salt and polyester acids with low molecular weight, unsaturated polyamine amide and polyester acids of low molecular weight	Hydro carbonate, ketenes, esters, alcohols, glycol ethers, glycol esters	less than 50 μm

Type of coating	Size of the $Mg(OH)_2$ Particles	Humidity in the initial hydroxide	Agent for the process of the “change of Agent”	Humidity of the Hydroxide before the diffusion	Dispersant for the diffusion	Solvent or thinner for the diffusion	Quality
Emulsion	1 to 190 nm	less than 5%	Aryl Ketenes, aryl alcohols, Glycol ester, glycol ether, etc..	less than 5%	Ammonium salt of an acrylic copolymer, salt of ethyl ammonium chloride a multifunctional polymer with an anionic character, sodium salt of an acrylic copolymer	Water	less than 20 µm

Type of coating		Size of the Mg(OH)$_2$ Particles	Humidity in the initial hydroxide	Agent for the process of the "change of Agent"	Humidity of the Hydroxide before the diffusion	Dispersant for the diffusion	Solvent or thinner for the diffusion	Quality
	UV	1 to 190 nm	less than 2%	Aryl Ketenes, aryl alcohols, Glycol ester, glycol ether, etc.	less than 2%	Copolymer with acidic groups, ethyl ammonium chloride salt of a polycarboxylic acid, ethyl ammonium chloride salt from a non saturated fatty acid, non saturated polyamine amide salt and polyester acids with low molecular weight, unsaturated polyamine amide and polyester acids of low molecular weight	Alcohols with 6 carbons or more, esters with 6 carbons or more, monomers	less than 15 μm
TEXTURI	Styrene-aryli	1 to 5 μm Acrylic-styrene	less than 5%	Water	less than 5%	Ammonium salt of an acrylic copolymer, salt of ethyl ammonium chloride a multifunctional polymer with an anionic character,	Water	less than 60 μm

Type of coating		Size of the $Mg(OH)_2$ Particles	Humidity in the initial hydroxide	Agent for the process of the "change of Agent"	Humidity of the Hydroxide before the diffusion	Dispersant for the diffusion	Solvent or thinner for the diffusion	Quality
ZED	c					sodium salt of an acrylic copolymer		
	Acrylic Vinyl	1 to 5 μm	less than 5%	water	less than 5%	Ammonium salt of an acrylic copolymer, salt of ethyl ammonium chloride a multifunctional polymer with an anionic character, sodium salt of an acrylic copolymer	water	less than 60 μm

For a better understanding of the use of the data contained in tables 1 and 2, the following examples are realized according to standards ASTM D1360. They offer illustrations of diverse alternatives for the creation of the additives according to the procedure of the present invention.

Example 1. Preparation of the additive to be used in a coating with an ethyl ammonium chloride base having a residual humidity of less than 5%.

1. Separate nanometric magnesium hydroxide with a humidity of 65% that has the following distribution of particle sizes: D_{10}, 59.0 nm; D_{50}, 92.7 nm; D_{90}, 153 nm; measured by laser ray diffraction in a machine marked COULTER LS 230. For the purpose of illustration, we will use 1200 grams.
2. Place the magnesium hydroxide in a 6 Liter recipient that has a pour spout.
3. Pour 3600 grams of ethylene glycol butyl ether, and agitate (dispersing) with a dispersing propellant to a peripheral velocity of between 5 and 30 m/ sec. This agitation (dispersion) is maintained for a period of between 5 and 15 minutes.

4. With the help of a centrifuge, a decanter, a filter, or other means of separating solids from liquids, as much of the liquid part as possible is removed, depending on the function of the equipment selected for completing the separation.
5. With the paste obtained, repeat steps 3 and 4 until the content of the water in said material is less than 5%.
6. The paste then from step 5 is placed in an appropriate drying machine and the organic solvent is eliminated. The dryer should not be operated at a temperature higher than the boiling point of the solvent, this guarantees that the power of magnesium hydroxide obtained does not suffer changes in the side of the particle and that its integration to the flame retarding additive will be efficient.
7. 590 grams of ethyl ammonium chloride will be placed into a 1.0 liter Berzelius glass without a spout.
8. The glass will be placed in a diffusion machine provided with a deflector and a propellant for the diffusion.
9. The resin is to be agitated at a moderate velocity: peripheral velocity of between 1 and 5 m/s.
10. Pour in ten grams of the dispersant DCM-305 (a copolymer with groups of acids), that is compatible with the resin.
11. Add to it 400 grams of the magnesium hydroxide that was obtained in point 6.
12. Maintain medium agitation until the magnesium hydroxide is completely humid.
13. Once the mix is homogenous, the velocity of the disperser is increased until it has enough cutting force that guarantees an efficient distribution: peripheral velocity between 15 and 30 m/s.
14. The diffusion is maintained during 15 minutes or the time necessary to reach the quality of the application.

Example 2. Preparation of the additive to be used in a coating with a nitrocellulosic base that has a required residual humidity of less than 5%.

1. Place 490 grams of the nitrocellulose resin in a 1.0 liter Berzelius glass without a spout.
2. Place the glass in a diffusion machine provided with a deflector and a propellant from the diffusion of 7 centimeters of diameter.
3. Agitate the resin at a moderate velocity.
4. Pour 10 grams of the dispersant DCM-305 (a copolymer with groups of acids), that is compatible with the resin.
5. Add in 400 grams of dry magnesium hydroxide with particle size being an average of 100 nanometers.
6. Maintain medium agitation until the magnesium hydroxide is completely humid.
7. Once the mix is homogenous, the velocity of the disperser is increased until it has enough cutting force that guarantees an efficient distribution: peripheral velocity between 15 and 30 m/s.

8. The diffusion is maintained during 15 minutes or the time necessary to reach the quality of the application.

The flame retarding additive for the ethyl ammonium chloride varnish, prepared in Example 1, can be mixed in different proportions with any ethyl ammonium chloride varnish that is found on the market. Also, the flame retarding additive for the nitrocellulose based sealer prepared in Example 2 can be mixed with any nitrocellulose based sealer that is on the market.

The most frequent is that in the varnish of an item made of wood it be used as much as a sealer as a varnish. Table 3 shows different combinations of sealers and varnishes mixed with the flame retarding additives prepared in Examples 1 and 2, and the results to summit them to the flame test according to the ASTM D1360 standard.

Table 3. Effect as the percentage of weight lost by the action of the flames, of the content of Mg(OH)2 in a nitrocellulose based sealer and a varnish with an ethyl ammonium chloride based resin both applied to a sample of wood.

		Test Number					
		1	**2**	**3**	**4**	**Control Group**	**Intumescent**
Sealer	**Type of resin**	Nitrocellulose	Nitrocellulose	Nitrocellulose	Nitrocellulose	Nitrocellulose	-
	Additive / Sealer	1 / 7	1 / 7	1 / 1	1 / 1	0	-
Varnish	**Type of resin**	Ethyl ammonium chloride	Ethyl ammonium chloride	Ethyl ammonium chloride	Ethyl ammonium chloride	Ethyl ammonium chloride	-
	Additive / Varnish	1 / 7	1 / 1	1 / 7	1 / 1	0	-
Loss of weight (%)		7.8	5.5	3.5	1.7	62.8	17.5

Table 4 shows the rest of the examples of the additives prepared with different types of resins that have applications to different types of coatings.

Table 4. Preparation of a flame retardant additive

Ex. #	Metallic Hydroxide	D_{50} mm	Inicial Humidity	Change of Vehicle	Final Humidity	Resin of the coating	Dispersant	Surfactant
1	$Mg(OH)_2$	0.0927	65%	ethylene glycol butyl ether	>5%	Ethyl ammonium chloride	Copolymer with acid groups	-
2	$Mg(OH)_2$	0.0927	<5%	-	<5%	Nitrocellulose	Copolymer with acid groups	-
3	$Mg(OH)_2$	0.0927	<5%	-	<5%	Phenolic	Copolymer with acid groups	Methyl hydrogyn polysiloxane
4	$Mg(OH)_2$	1.102	<5%	-	<5%	Polyurethane	Copolymer with acid groups	Methyl hydrogyn polysiloxane
5	$Mg(OH)_2$	1.102	55%	water	>5%	Vinyl	Ammonium salt made of an acrylic copolymer	Methyl hydrogyn polysiloxane
6	$Mg(OH)_2$	1.102	55%	water	>5%	Acrylic vinyl	Ammonium salt made of an acrylic copolymer	Methyl hydrogyn polysiloxane
7	$Mg(OH)_2$	1.102	<5%	-	<5%	Ethyl ammonium chloride	Copolymer with acid groups	Methyl hydrogyn polysiloxane

Ex. #	Metallic Hydroxide	D_{50} mm	Inicial Humidity	Change of Vehicle	Final Humidity	Resin of the coating	Dispersant	Surfactant
8	$Mg(OH)_2$	1.102	55%	water	>5%	Ethyl ammonium chloride 0-VOC	Ammonium salt made of an acrylic copolymer	Methyl hydrogyn polysiloxane

Table 4. Preparation of a flame retardant additive (continued)

Ex. #	Metallic Hydroxide	D_{50} mm	Inicial Humidity	Change of Vehicle	Final Humidity	Resin of the coating	Dispersant	Surfactant
9	$Mg(OH)_2$	1.102	<5%	-	<5%	Acrylic epoxy catalyzed by UV	Copolymer with acid groups + non saturated polyamine amide salt and polyester acids with low molecular weight	Methyl hydrogyn polysiloxane
10	$Mg(OH)_2$	1.102	55%	water	>5%	Acrylic – styrene	Ammonium salt made of an acrylic copolymer	Methyl hydrogyn polysiloxane
11	$Al(OH)_3$	6.86	>5%	-	<5%	Ethyl ammonium chloride	Copolymer with acid groups	Methyl hydrogyn polysiloxane
12	$Al(OH)_3$	6.86	40%	water	>5%	Vinyl	Ammonium salt made of an acrylic copolymer	Methyl hydrogyn polysiloxane

Ex. #	Metallic Hydroxide	D_{50} mm	Inicial Humidity	Change of Vehicle	Final Humidity	Resin of the coating	Dispersant	Surfactant
13	$Al(OH)_3$	6.86	<5%	-	<5%	Polyurethane	Copolymer with acid groups	Methyl hydrogyn polysiloxane
14	$Al(OH)_3$	6.86	40%	water	>5%	Ethyl ammonium chloride 0-VOC	Ammonium salt made of an acrylic copolymer	Methyl hydrogyn polysiloxane

The effects of the use of the additive with magnesium hydroxide in the wood coatings in the previous tests are illustrated in Table 5. They show the result of the examples of the application of the prepared additive according to the previously described procedure.

The effect of the flame retardant when the coating includes magnesium hydroxide can be clearly observed. In the physical tests, it can be seen that that there is no change in the final appearance of the coating with the additive of the invention after its application with respect to the appearance that the coating offers without the additive; this is also valid for coatings that have a high transparency such as varnishes.

Table 5. Effect of the flame retarding additive, as a percentage of the weight loss, applied in a sample of wood according to the ASTM D1360 standard.

Sealer		**Coating**		**Metallic Dioxide**	**Weight Loss (%)**
Type of Sealer	**Additive / Sealer**	**Type of Coating**	**Additive / Coating**		
Nitrocellulose	1/7	Ethyl ammonium chloride varnish	1/7	$Mg(OH)_2$	**7.8**
Nitrocellulose	1/7	Phenolic Varnish	1/7	$Mg(OH)_2$	**3.6**
Nitrocellulose	1/1	Ethyl ammonium chloride varnish	1/1	$Al(OH)_3$	**14.3**
-	-	Intumescent	-	-	**17.5**
Nitrocellulose	-	Ethyl ammonium chloride varnish	-	-	**62.8**
Nitrocellulose	-	Phenolic Varnish	-	-	**66.1**

-	-	Ethyl ammonium chloride varnish 0-VOC	1/1	$Mg(OH)_2$	**6.7**
-	-	Varnish of Acrylic epoxy catalyzed by UV	1/1	$Mg(OH)_2$	**30.1**
-	-	Polyurethane varnish	1/1	$Mg(OH)_2$	**11.4**
-	-	Ethyl ammonium chloride varnish 0-VOC	-	-	**61.3**
-	-	Varnish of Acrylic epoxy catalyzed by UV	-	-	**71.0**

Table 5. Effect of the flame retarding additive, as a percentage of the weight loss, applied in a sample of wood according to the ASTM D1360 standard. (continued)

Sealer		**Coating**		**Metallic Dioxide**	**Weight Loss (%)**
Type of Sealer	**Additive / Sealer**	**Type of Coating**	**Additive / Coating**		
-	-	Polyurethane varnish	-	-	**72.4**
-	-	Ethyl ammonium chloride enamel	1/1	$Mg(OH)_2$	**2.8**
-	-	Ethyl ammonium chloride enamel	-	-	**8.2**
-	-	Acrylic Styrene water- proofer	1/1	$Mg(OH)_2$	**10.7**
-	-	Acrylic Styrene water -proofer	-	-	**58.4**
-	-	Acrylic Vinyl Paint	1/1	$Mg(OH)_2$	**5.2**
-	-	Vinyl Paint	1/1	$Mg(OH)_2$	**6.0**

-	-	Acrylic Vinyl Paint	-	-	**52.6**
-	-	**Vinyl Paint**	-	-	**52.6**

In this aspect, the size of the hydroxide particles is important, as well as the uniformity of the size and the homogeny of the distribution of the particles in the final layer of the coating once applied.

As has been described and illustrated through the previous examples, the present invention is directed to the preparation of an additive that has the properties of a flame retardant and a suppresser of smoke, without affecting the properties of the coating to which it is added.

In the examples, there has been used some common coatings for the protection of surfaces, in particular wood and its derivative, as can be seen by a technician with knowledge of the art, these examples do not limit the reach of the invention but they are to be illustrative of the same , and the use of the additive in other coatings for the application to other surfaces, is found to be also within the reach of the present invention that, thus should be considered in light of the following claims.

CLAIMS

Once the preferred modes are described of the invention, what should be considered new and thus part of the claim of this property is:

1. A procedure for the preparation of a flame retarding additive to be incorporated in conventional coatings to protect surfaces that are susceptible to combustion. The additive has a base of a metallic hydroxide as the flame retarding agent and smoke suppresser, said procedure encompasses the following stages of (a) pretreatment of the flame retarding agent and (b) preparation of the additive, said procedure is characterized as such because in stage (a) of the pretreatment, the humidity contained in the metallic hydroxide is replaced by a compatible agent with the dilutant of the final application, through the washing of the initial hydroxide with the compatible agent.
2. A procedure for the preparation of a flame retarding additive according to the first claim is characterized because the wash is realized with strong agitation.
3. A procedure for the preparation of a flame retarding additive according to the second claim is characterized because the machine for the agitation generates a peripheral velocity of between 5 m/s and 30 m/s.
4. A procedure for the preparation of a flame retarding additive according to the first claim is characterized because the agitation is maintained for between 5 and 30 minutes.
5. A procedure for the preparation of a flame retarding additive according to the first claim is characterized because after the agitation, the separation of the phases is allowed, as well as the elimination of the liquid phase.
6. A procedure for the preparation of a flame retarding additive according to the first claim is characterized because the washing is repeated until a residual humidity of 5% in the solid stage is obtained.
7. A procedure for the preparation of a flame retarding additive according to the first claim is characterized because the vehicle for the washing is selected between the solvents or the thinners used in conventional coatings in a way that will be compatible with the solvent of the thinner of the objective coating.
8. A procedure for the preparation of a flame retarding additive according to the seventh claim is characterized because the vehicle for the washing is the same as the solvent or the thinner of the objective coating.
9. A procedure for the preparation of a flame retarding additive according to the first claim is also characterized because if the metallic hydroxide is susceptible to react with the selected vehicle for the wash or the resin of the coating, the metallic hydroxide is submitted to a surface treatment with surfactants before the wash.
10. A procedure for the preparation of a flame retarding additive according to the first claim is characterized because when the residual humidity allowed for the objective coating is close to zero, the product obtained from the "change of agent" is summated to a drying process.
11. A procedure for the preparation of a flame retarding additive according to the tenth claim is characterized because the temperature of the operation in said drying stage should be inferior to the boiling point of the agent.

12. A procedure for the preparation of a flame retarding additive according to the eleventh claim is characterized because it remains in the drying stage until obtaining the residual humidity tolerated by the objective coating.
13. A procedure for the preparation of a flame retarding additive according to the first claim is characterized because the metallic hydroxide obtained in the first stage of the pretreatment contains a humidity of between 0% and 5% and is dispersible in a compatible resin with the objective coating.
14. A procedure for the preparation of a flame retarding additive according to the thirteenth claim is characterized because said metallic hydroxide is able to be stored for prolonged periods of time, until the time it is needed to prepare the dispersion that forms the flame retarding additive.
15. A procedure for the preparation of a flame retarding additive according to the first claim is characterized because the second phase of the procedure consists of a diffusion of a metallic hydroxide of low humidity with a resin and a dispersant.
16. A procedure for the preparation of a flame retarding additive according to the fifteenth claim is characterized because what is added to the second phase of the procedure is a metallic hydroxide dispersible in the resin of the objective coating with humidity content of between 0% and 5%, being the maximum tolerated by the objective coating.
17. A procedure for the preparation of a flame retarding additive according to the sixteenth claim is characterized because the resin is a resin that is compatible with the objective coating.
18. A procedure for the preparation of a flame retarding additive according to the fifteenth claim is characterized because the dispersant is compatible with that of the objective coating.
19. A procedure for the preparation of a flame retarding additive according to the fifteenth claim is characterized because the dispersion is done with a peripheral velocity of between 15 and 30 m/s.
20. A procedure for the preparation of a flame retarding additive according to the fifteenth claim is characterized because the percent of the dispersant in the mix is maintained between 0.5% and 10% in relation to the hydroxide in the dry base.
21. A procedure for the preparation of a flame retarding additive according to the fifteenth claim is characterized because the product obtained is a flame retarding additive that contains from 5% to 99% of the weight of metallic hydroxide.
22. A flame retarding additive is incorporated in conventional coatings to protect surfaces susceptible to combustion, the additive is obtained by the procedure that claim one confirms, containing a metallic hydroxide as a flame retarding agent and a smoke suppresser, an agent, a dispersant and a resin, said additive is characterized because:
    a. The metallic hydroxide has an average particle size in the range of 1 nanometer to 10 microns
    b. The agent is compatible with the coating for which the additive was prepared
    c. The resin is compatible with the coating for which the additive was prepared
    d. The dispersant is compatible with the objective coating.

23. A flame retarding additive is incorporated in conventional coatings according to the 22nd claim characterized by the fact that the metallic hydroxide is found to be in a proportion from 5% to 99% of the weight.
24. A flame retarding additive is incorporated in conventional coatings according to the 22nd claim, characterized by the fact that the dispersant is found to be present in a quantity of between 0.5% and 10% in the base of the hydroxide in the dry base.
25. A flame retarding additive is incorporated in conventional coatings according to the 22nd claim, characterized by the fact that the additive has a humidity content equal or less than that tolerated by the objective coating.
26. A flame retarding additive is incorporated in conventional coatings according to the 22nd claim, characterized by the fact that the metallic hydroxide is preferably magnesium hydroxide.
27. A flame retarding additive is incorporated in conventional coatings according to the 22nd claim, characterized by the fact that said additive is homogenously dispersed in the objective coating once it is incorporated into the same.
28. A flame retarding additive is incorporated in conventional coatings according to the 22nd claim, characterized by the fact that said additive does not interfere with the desirable properties of the coating for which it is destined, such as the shine, transparency, the covering power, or the mechanical resistance.
29. A flame retarding additive is incorporated in conventional coatings according to the 22nd claim, characterized by the fact that said additive presents a grain quality similar to that of the coating in which it is included.
30. A flame retarding additive is incorporated in conventional coatings according to the 22nd claim, characterized by the fact that the size of the magnesium hydroxide particles are found to be in the range from 1 to 190 nanometers for the coating that requires transparency in its finish.
31. A flame retarding additive is incorporated in conventional coatings according to the 22nd claim, characterized by the fact that the size of the magnesium hydroxide particles are found to be in the range of 1 to 10 microns for the coatings that are opaque or texturized.

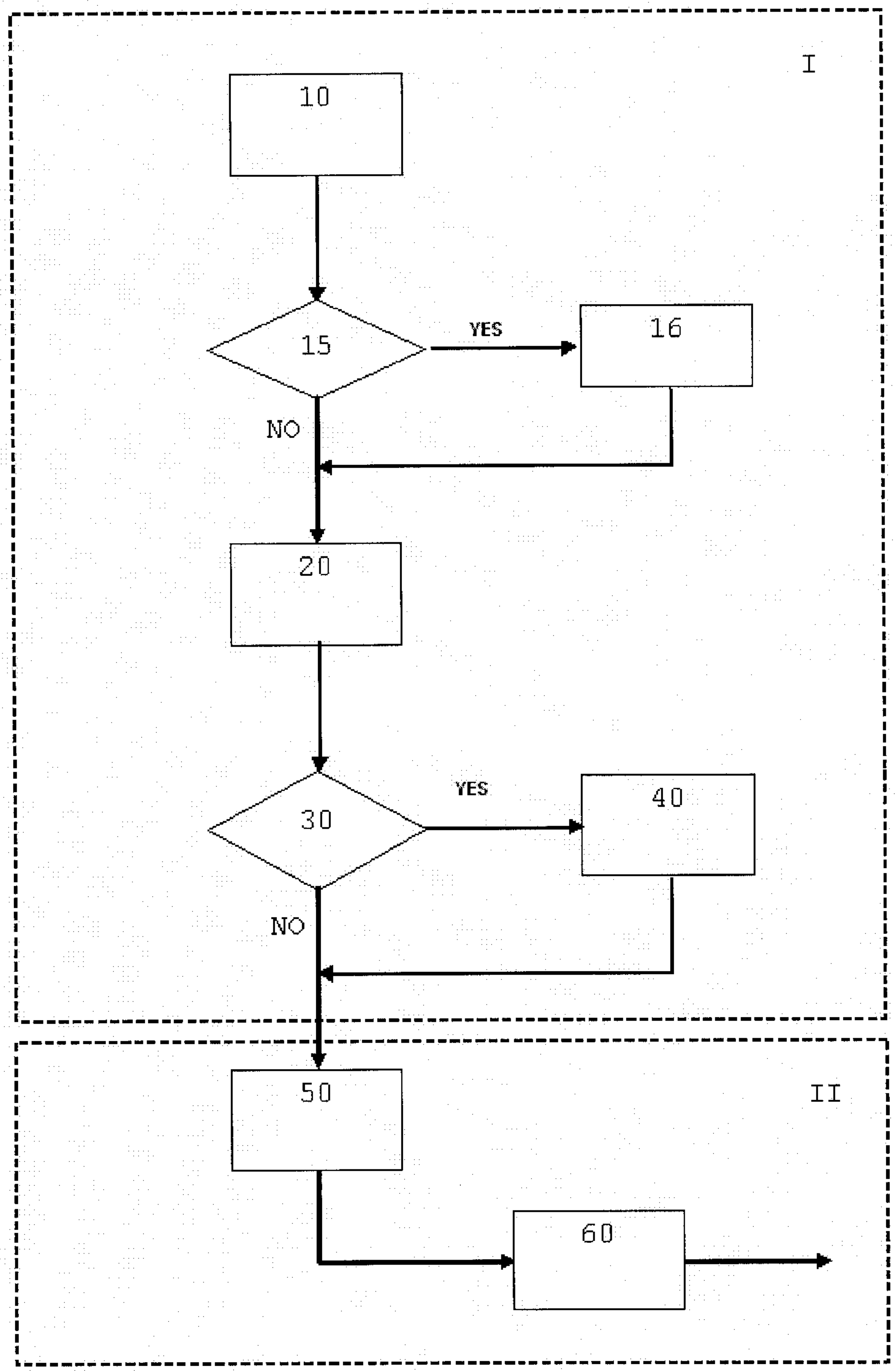
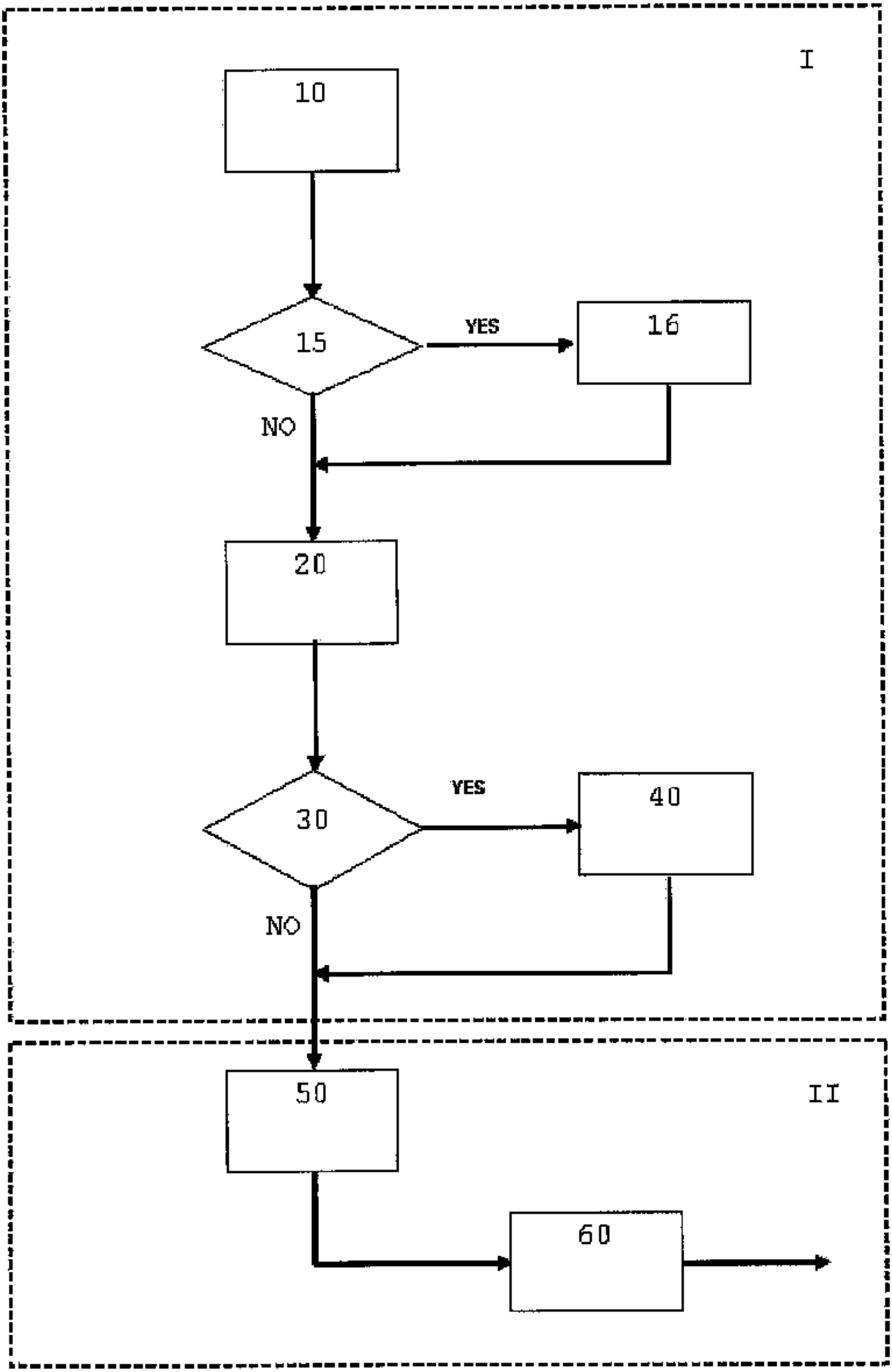


FIGURE 1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10
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30
YES
40
NO
50
II
60