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(54) FIRE RETARDANT RESINOUS COMPOSITIONS

(71) We, KUREHA KAGAKU KOGYO KABUSHIKI KAISHA, a company organised and existing under the laws of Japan, of 8, Nihonbashi Horidome-Cho, 1-Chome, Chuo-Ku, Tokyo-To, Japan, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a novel ABS resin composition of improved fire retardance and workability. More particularly, this invention relates to a resinous composition comprising a blend mixture of an ABS resin and either a copolymer resin consisting essentially of vinyl chloride and a higher alkyl vinyl ether or a vinyl chloride resin component containing the copolymer.

ABS resins (acrylonitrile-butadiene-styrene resins) are of easy combustibility, and a fire-retarded ABS resin has been keenly needed.

ABS resins have been generally incorporated with fire retardant additives such as halogen-containing compounds and phosphorus-containing compounds, if desired, together with fire retardant auxiliaries such as antimony trioxide.

Especially, vinyl chloride resins (hereinafter referred to as PVC) have been widely employed as a fire retardant for ABS resins, since PVC is readily blended with ABS resins and does not cause a decrease in mechanical strength, and bleeding and weathering after a long time of use, unlike the case where low-molecular retardant additives such as halogen compounds are employed.

As noted above, PVC is an excellent fire retardant for ABS resin, but there remain some processing problems owing to the difference in heat stabilities between PVC and ABS resin. That is, PVC is inferior in heat stability to ABS resin. When a blend of PVC and ABS resin is moulded at a temperature suitable for processing ABS resin, PVC undergoes thermal decomposition. On the other hand, when the blend is processed at a lower temperature suitable for PVC, the processing of the blend is extremely difficult because of its very high melt viscosity.

Addition of plasticizers such as dioctyl phthalate and dibutyl phthalate has been proposed so as to eliminate these defects, but this method results in a decrease in heat stability of the blend as well as deterioration of fire retardance and a great decrease in the stiffness inherent in ABS resin compositions.

We have sought to eliminate these defects in PVC employed as a fire retardant for ABS resin. We have employed a copolymer resin of a higher alkyl vinyl ether and vinyl chloride or a blend thereof with polyvinyl chloride in place of PVC in ABS resin compositions, and have found that the resulting resinous compositions has a satisfactory flow property even at a temperature suitable for processing PVC resin in the same degree as ABS resins, and exhibits an excellent heat distortion temperature.

The invention thus provides a high-impact resinous composition which is fire retardant and has excellent workability or processability.

Other features and advantages of the invention will appear more fully from the following description.

In accordance with the present invention, there is provided a resinous composition comprising a blend of (1) 80 to 30 parts by weight of an ABS resin and (2) 20 to 70 parts by weight of a vinyl chloride resin component comprising (i) 100 to 5 parts by weight of a copolymer resin of 0.5 to 40% by weight of a higher alkyl vinyl ether having 12 to 18 C-atoms in the alkyl group, and 99.5% to 60% by weight of vinyl chloride, and (ii) 0 to 95 parts by weight of a chloride polymer resin other than that defined under (i).

The resinous composition of the present

invention has a good flow property and fire retardance and can be advantageously utilized substantially for the same purposes as conventional ABS resin compositions are employed.

(1) Components of the resinous compositions

(a) ABS Resin

The ABS Resin, one of the main components of the present composition, is basically a ternary polymer comprising polymerized acrylonitrile, or, more broadly an olefinically unsaturated nitrile, butadiene and styrene or, more broadly, and ethylenically unsaturated aromatic compound, containing, if desired, a further monomer component such as methyl methacrylate.

Conventional ABS resins are products of the polymerization of acrylonitrile and styrene, or, more broadly, an ethylenically unsaturated aromatic compound, in the presence of a butadiene-based rubber polymer such as polybutadiene or a copolymer of butadiene and styrene (SBR). In the present invention, however, it should be understood that the term "ABS resin" encompasses, in addition to the conventional ABS resins, a polymeric mixture of (1) a product of the polymerization of an ethylenically unsaturated aromatic compound such as styrene or an olefinically unsaturated nitrile such as acrylonitrile in the presence of a butadiene based rubber polymer such as polybutadiene or a copolymer of α-butadiene and styrene (SBR), and (2) a copolymer of an ethylenically unsaturated aromatic compound and an olefinically unsaturated nitrile.

A part of the styrene can be replaced by another ethylenically unsaturated aromatic compound such as α-methylstyrene or a vinyl toluene, and the acrylonitrile can be partly replaced by a methacrylic ester such as methyl methacrylate. Moreover, a part or all of the acrylonitrile may be replaced by methacrylonitrile.

The ABS resin to be generally employed in the present invention comprises 10 to 40 parts by weight of a butadiene rubber component, 80 to 25 parts by weight of an ethylenically unsaturated aromatic compound, and 35 to 5 parts by weight of acrylonitrile. As to the butadiene rubber component, polybutadiene and/or copolymers consisting of a major part of butadiene and a minor part of a mono-olefinic monomer copolymerizable therewith such as styrene, acrylonitrile or vinylidene chloride are employed generally.

The process for preparation of the ABS resin *per se* does not comprise any part of the present invention, and the resin is generally produced either by graft polymerizing an aromatic vinyl and acrylonitrile (and/or methacrylonitrile) onto a butadiene polymer rubber, or graft polymerizing some parts of the grafting monomer components onto the rubber, and then blending the resulting graft polymer with an ethylenically unsaturated aromatic compound-acrylonitrile copolymer.

In a preferred embodiment, the ABS resin comprises (a) a graft polymer consisting of a graft polymer of acrylonitrile and styrene onto a butadiene-based polymer, a graft polymer of methyl methacrylate and styrene onto a butadiene-based polymer or a graft polymer of acrylonitrile, styrene and methyl methacrylate onto a butadiene-based polymer, and (b) a copolymer comprising polymerized acrylonitrile and styrene, the term "graft polymer" being used as defined herein. The term "graft polymer" as used herein means a product of polymerisation of a monomer such as styrene and/or acrylonitrile in the presence of a polymer such as a butadiene-based polymer irrespective of whether the product is a true *graft* polymer or not.

(b) Polyvinyl chloride resin component

The vinyl chloride resin compound to be employed as a blending polymer in the present invention is (1) a copolymer resin consisting essentially of 0.5 to 40% by weight of a higher alkyl vinyl ether and 99.5 to 60% by weight of vinyl chloride (hereinafter referred to as HCA), or (2) a blend of less than 100% to 5% by weight of the HCA and up to 95%, viz. 0 (not inclusive) to 95%, by weight of a vinyl chloride polymer resin (preferably, vinyl chloride homopolymer resin). Such vinyl chloride resin component is described in detail in Japanese Patent Publication No. 22733/1961.

When the copolymer resin contains less than 0.5% by weight of a higher alkyl vinyl ether, the resulting resinous composition fails to have a sufficient flow property. When the copolymer resin contains more than 40% by weight of a higher alkyl vinyl ether, it has poor compatability with the ABS resin and the resulting resinous composition fails to have good mechanical properties. In the same way, the amount of a higher alkyl vinyl ether component contained in the vinyl chloride resin compound is desirably not less than 0.5% by weight of the compound.

Higher alkyl vinyl ethers the alkyl group of which contains 12 to 18 carbon atoms are generally employed in the present invention. When an alkyl vinyl ether having an alkyl group of less than 12 carbon atoms

is used, the resulting resinous composition is inferior in workability. An alkyl vinyl ether having more than 18 carbon atoms is not suitable since its copolymerizability with vinyl chloride is poorer. Suitable alkyl vinyl ethers are exemplified by lauryl, cethyl, stearyl, myristyl and like vinyl ethers. These copolymer resins are prepared by a conventional suspension or emulsion polymerization.

In one aspect of the present invention, the vinyl chloride resin component comprises a blend of a vinyl chloride polymer resin in a quantity up to 95% by weight of the blend. The vinyl chloride polymer resin can be any of the resinous copolymers of vinyl chloride and a monomer other than the higher alkyl vinyl ether, but is preferably a homopolymer of vinyl chloride.

(c) Impact modifiers

An impact modifier of the kind employed in conventional vinyl chloride resins can further be incorporated into the resinous compositions of the present invention.

While ABS resins have excellent impact resistance, it is noted that, in the resinous composition of the present invention, the flow property in processing increases but impact resistance is lowered as the ratio of HCA contained in the vinyl chloride resin compound, and the ratio of alkyl vinyl ether contained in the HCA become larger. The impact resistance of the present resinous composition can be advantageously improved by the addition of a small amount of a conventional impact modifier without impairing its good processability. The amount of the impact modifier to be employed in the present resinous composition varies in accordance with the vinyl chloride resin compound to be employed. The impact modifier is generally used in an amount of not more than about 20 parts by weight relative to 100 parts by weight of the present resinous composition.

The impact modifiers for vinyl chloride resins are exemplified by MBS resin (a terpolymer resin of methyl methacrylate, butadiene and styrene, especially a butadiene rubber onto which monomer components comprising methyl methacrylate and styrene are graft polymerized), a chlorinated polyethylene, and an ethylene-vinyl acetate copolymer rubber onto which vinyl chloride is graft polymerized. The impact modifiers for PVC on the market can be generally employed.

(d) Other additives

As other additives, an antimony compound which is an effective fire retardant auxiliary such as antimony oxide, antimony trisulfide or triphenyl antimony can be used alone or in combination with other ordinary fire retardant additives.

The fire retardant additives generally employed in resinous compositions are exemplified by chlorinated paraffins, hexabromobenzene, tetrabromoethane, tetrabromobisphenol A, and the like.

Suitable amounts of heat stabilizers, lubricants and/or pigments which are generally used in PVC compounds can also be employed upon processing the resinous composition of the present invention.

The invention is further explained by way of the following Examples, which are illustrative only and are not limitative.

Examples 1—10 and
comparative examples 1—7

In these examples, vinyl chloride cetyl vinyl ether copolymers containing 10% and 5% of cetyl vinyl ether supplied by Kureha Kagaku Kogyo, Japan, under the trade name HCA, and homopolymers of vinyl chloride having a relatively good flow property supplied by Kureha Kagaku Kogyo under the trade name S9006 (degree of polymerization 600) and S9007 (degree of polymerization 700) were employed. They were blended with a commercially available ABS resin comprising 20% of acrylonitrile, 25% of butadiene and 55% of styrene on the weight basis, to obtain resinous compositions.

One part by weight of organo-tin maleate stabilizer (N2000E supplied by Nitto Chemical Industry Co., Japan) was added to 100 parts by weight of each of the resinous compositions, followed by kneading the mixture on a test roll having a surface temperature of 150° C to prepare sheets. The sheets were formed into test pieces by press moulding.

Fire retardant tests were carried out in accordance with ASTM-D-635. The results were classified into "combustible", "slow-burning" and "incombustible" were evaluated as "pass" and "combustible" as "failure". Impact strength (Izod impact strength) and heat distortion temperature were measured according to JIS (Japanese Industrial Standard)-6871. Flow property (fluidity) was measured on an extruded amount of the sample resin by means of a Koka flow tester (supplied by Shimazu Seisakusho, Japan) with a load of 150 kg/cm² and a nozzle 0. cm × 10 cm and at a temperature of 200°C. The properties of the resulting resinous compositions are shown in the following Table 1.

In Table 1, the underlined numerals denote the values which do not satisfy the standards of the present resinous composition. A resinous composition which satisfies fire retardance, impact resistance, heat stability and flow property simultaneously can be readily obtained according to the present invention by

blending ABS resins with the vinyl chloride resin compounds. When HCA is not employed, the composition is inferior in flow property (cf. comparative examples 4 and 5). The flow property itself may be enhanced by the addition of a plasticizer such as DOP (dioctyl phthalate) to the composition, but heat distortion temperature is markedly lowered (cf. comparative examples 6 and 7). The data of the examples are clearly superior to those of the comparative examples.

The mixed resin compositions in which the amounts of vinyl chloride resin compounds are less than 20% by weight are not satisfactory in the degree of fire retardance (cf. comparative examples 1 and 2). The mixed resin compositions in which less than 30% by weight of ABS resins are contained are inferior in both impact strength and heat distortion temperature (cf. comparative example 3).

TABLE 1

Examples No.		Compositions (parts by weight) ABS	HCA	PVC(1)	PVC(2)	Properties of the Resinous Compositions: Degree of Fire Retardance (ASTM–D–635)	Impact Strength (JIS–6871) (kg cm/cm)	Heat Distortion Temperature (JIS–6871) (°C. 18.5 kg/cm²)	Fluidity (c.c/sec × 10^{-2})
Examples of the Invention	1	40	30	30	–	incombustible (pass)	25	72	6.2
	2	50	25	25	–	– ,, – (,,)	36	74	5.9
	3	60	20	20	–	– ,, – (,,)	40	76	4.5
	4	70	15	15	–	slow-burning (,,)	30	79	4.4
	5	80	10	10	–	slow-burning (,,)	27	83	4.2
	6	50	10	–	40	incombustible (pass)	61	77	3.2
	7	50	20	–	30	– ,, – (,,)	58	75	3.5
	8	50	30	–	20	– ,, – (,,)	35	73	4.9
	9	50	40*	–	10	– ,, – (,,)	25	75	5.3
	10	50	50*	–	–	– ,, – (,,)	20	73	5.9
Comparative Examples	1	100	–	–	–	combustible *(failure)*	42	85	*2.5*
	2	9(	5	–	5	– ,, – *(failure)*	28	84	3.0
	3	20	40	40	–	incombustible (pass)	*5*	69	5.8
	4	50	–	50	–	incombustible (pass)	62	79	*2.4*
	5	50	–	–	50	– ,, – (,,)	76	79	*1.9*
	6**	50	–	–	50	incombustible (pass)	68	*63*	*2.3*
	7**	50	–	–	50	– ,, – (,,)	55	*58*	5.6

Note: (a) The underlined values denote that the values are not satisfactory.
(b) * a vinyl chloride copolymer containing 5% of cetyl vinyl ether was used.
(c) PVC(1) and PVC(2) stand for PVC having degrees of polymerization of 600 and 700, respectively.
(d) ** The resin compositions of comparative examples 6 and 7 were prepared respectively by adding 6 parts (C. Ex. 6) or 10 parts (C. Ex. 7) by weight of DOP to the composition of comparative example 5.

Examples 11—13

These examples show in the following Table 2 the effects obtained by incorporating in the resinous compositions of Examples 9 and 10, an MBS resin (BTA IIIs supplied by Kureha Kagaku Kogyo) sold as an impact modifier for vinyl chloride resins or a commercially available chlorinated polyethylene ClPE).

The impact strength of the resinous composition can readily be enhanced without impairing fire retardance, heat stability and fluidity thereof, as shown in the Table.

TABLE 2

Example No.		Composition (parts by weight)		Properties of the Resinous Compositions			
		**	BTA IIIs	Impact Strength JIS–6871 (kg cm/cm)	Fire Retardance ASTM–D–635	Heat Distortion Temperature JIS–6871 (°C)	Fluidity (c.c./sec × 10^{-2}) at 200°C
Examples of Invention	9	100	0	25	incombustible (pass)	75	5.3
	11	100	5	38	– ,, – (,,)	74	5.0
	12	100	10	45	– ,, – (,,)	72	4.9
		***	ClPE*				
	10	100	0	20	– ,, – (,,)	73	5.9
	13	100	5	35	– ,, – (,,)	71	6.1

Note: * ClPE stands for chlorinated polyethylene having a degree of chlorination of 35%.

** The resinous composition of Example 9.

*** The resinous composition of Example 10.

Examples 14—17 and Comparative Examples 8—10

The resinous composition of Example 4 was mixed with ordinary low-molecular fire retardant additives, namely antimony trioxide (Sb_2O_3) or hexabromobenzene (C_6Br_6). The results are shown in Table 3 below. The fire retardant effect was further exhibited by the addition of a small amount of the fire retardant additive to the resinous composition of the present invention. Moreover, the fire retardant additive gave no substantially adverse effect to the other properties of the resinous composition.

On the other hand, a mixture of ABS resin alone and a small amount of the fire retardant additive (Sb_2O_3) scarcely exhibited a fire retardant effect, and the impact strength thereof was markedly poorer. The results are also shown in Table 3 (comparative examples 8, 9, and 10).

TABLE 3

Example No.		Composition (parts by weight)		Properties of the Resinous Composition	
		Resin comp. of Example 4	Fire retardant additives	Fire retardance ASTM–D–635	Impact Resistance JIS–6871 (kg. cm/cm)
Examples of the Invention	4	100	0	slow-burning (pass)	30
	14	100	2 (Sb_2O_3)	incombustible (pass)	26
	15	100	4 (Sb_2O_3)	– ,, – (,,)	22
	16	100	2 (C_6Br_6)	– ,, – (,,)	29
	17	100	4 (C_6Br_6)	– ,, – (,,)	28
Comparative Examples		ABS Resin*	Fire retardant additive		
	8	100	0	combustible (failure)	28
	9	100	10 (Sb_2O_3)	– ,, – (,,)	14
	10	100	15 (Sb_2O_3)	– ,, – (,,)	9

Note: * The ABS resin comprises 22 % of acrylonitrile, 23 % of butadiene and 50 % of styrene.

Examples 18—20

Combinations of the following graft polymers prepared by polymerizing styrene, acrylonitrile and methylmethacrylate on a butadiene-based polymer, and a styrene-acrylonitrile copolymer (AS resin) were chosen as ABS resins to be used in the present invention.

Graft Polymer (1):

To a latex containing 65 parts by weight of a copolymer rubber with an average particle size of 0.3μ, consisting of 90% by weight of butadiene and 10% by weight of styrene, were added a monomer mixture consisting of 27 parts by weight of styrene containing 0.035 part by weight of diisopropylbenzene hydroperoxide and 0.075 part by weight of divinylbenzene and 8 parts by weight of acrylonitrile, and 10 parts by weight of distilled water containing 0.0175 part by weight of formaldehyde sodium sulfoxylate. Then, polymerization was carried out continuously for 7 hours at a temperature of 60°C.

To the latex thus obtained were added 0.5 part by weight each of ditertiarybutyl paracresol and dilaurylthiodipropionate, and thereafter the latex was subjected to coagulation with an aqueous solution of hydrochloric acid, washed with water, dehydrated and dried at 60°C, whereupon a graft polymer (ABS resin) was obtained in powder form. The polymerization yield was approximately 100%.

Graft Polymer (2):

Graft polymer (2) (MBS resin) was produced in the same manner as graft polymer (1) except that 27 parts by weight of styrene and 8 parts by weight of acrylonitrile polymerized on the rubber polymer in graft polymer (1) were replaced by 17.5 parts by weight of styrene and 17.5 parts by weight of methyl methacrylate.

Graft Polymer (3):

Graft polymer (3) (ABSM resin) was produced in the same manner as graft polymer (1) except that 27 parts by weight of styrene and 8 parts by weight of acrylonitrile polymerized on the rubber polymer in graft polymer (1) were replaced by 13.5 parts by weight of styrene, 17.5 parts by weight of methyl methacrylate and 4 parts by weight of acrylonitrile.

AS Resin:

The following constituents were charged into a reaction vessel and after replacement of the atmosphere therein with nitrogen, polymerization was carried out continuously for 20 hours at a temperature of 60°C.

	Parts by weight
styrene	75
acrylonitrile	25
tertiarydodecylmercaptan	0.25
azobisisobutyronitrile	0.25
polyethylene oxide	0.3
methylcellulose	0.1
distilled water	100

The slurry obtained was dehydrated by a centrifugal separator, washed with water and dried at 80°C., whereupon a copolymer was obtained in bead form.

The reduced viscosity (η sp/C) of this copolymer in dimethyl formamide at a concentration of 0.4 g/dl at 30°C. was 0.8 g/dl.

The graft polymers and the AS resin thus prepared were each blended with polyvinyl chloride compound in a dry state to produce resinous compositions.

A stabilizer was added and samples were prepared in the same manner as Example 1, the samples being used in the respective tests.

The properties of these resinous compositions are summarized in Table 4 as follows:

TABLE 4

Example No.	Composition (Parts by weight)				Properties of the Resinous Composition			
	Graft Polymer	AS	HCA*4	PVC*5	Fire retardance (ASTM–D–635)	Impact Resistance (JIS–6871)	Thermal Distortion Temperature (JIS–6871)	Fluidity (c.c./sec×10^{-2})
18	15*1	35	20	30	incombustible (pass)	75 kg. cm/cm^2	76°C	3.7
19	15*2	35	20	30	,,	47 ,,	75°C	3.8
20	15*3	35	20	30	,,	63 ,,	75°C	4.1

*1 Graft polymer (1) (ABS resin)

*2 Graft polymer (2) (MBS resin)

*3 Graft polymer (3) (ABSM resin)

*4 Copolymer of vinyl chloride containing 10% by weight of cetylvinyl ether

*5 Polyvinyl chloride resin having a degree of polymerization of 600

Table 2 shows that the four resin mixtures prepared by blending graft polymers and AS resin with polyvinyl chloride compositions demonstrate the advantages of the present invention, i.e., low combustibility, high impact strength, heat resistance and fluidity under processing conditions.

While the invention has been particularly shown and described with reference to preferred embodiments thereof, it will be understood by those skilled in the art that the foregoing and other changes can be made without departing from the scope of the invention.

WHAT WE CLAIM IS:—

1. A resinous composition comprising a blend of (1) 80 to 30 parts by weight of an ABS resin and (2) 20 to 70 parts by weight of a vinyl chloride resin component comprising (i) 100 to 5 parts by weight of a copolymer resin of 0.5 to 40% by weight of a higher alkyl vinyl ether having 12 to 18 C-atoms in the alkyl group, and 99.5% to 60% by weight of vinyl chloride, and (ii) 0 to 95 parts by weight of a vinyl chloride polymer resin other than that defined under (i).

2. A resinous composition as set forth in claim 1, in which said ABS resin comprises

polymerized butadiene, acrylonitrile and/or methacrylonitrile, and styrene.

3. A resinous composition as claimed in claim 2, in which the ABS resin additionally comprises one or more other ethylenically unsaturated aromatic compounds and one or more methacrylates.

4. A resinous composition as claimed in claim 1, in which said ABS resin comprises 10 to 40 parts by weight of a butadiene rubber component, 80 to 25 parts by weight of an ethylenically unsaturated aromatic compound including styrene, and 35 to 5 parts by weight of acrylonitrile and/or methacrylonitrile.

5. A resinous composition as claimed in claim 4, in which said butadiene rubber component is polybutadiene and/or a copolymer comprising a major part of butadiene and a minor part of one or more monoolefinic monomers.

6. A resinous composition as claimed in claim 1, in which said ABS resin comprises (a) a graft polymer consisting of a graft polymer of acrylonitrile and styrene onto a butadiene-based polymer, a graft polymer of methyl methacrylate and styrene onto a butadiene-based polymer or a graft polymer of acrylonitrile, styrene and methyl methacrylate onto a butadiene-based polymer, and (b) a copolymer comprising polymerized acrylonitrile and styrene, the term "graft polymer" being used as defined herein.

7. A resinous composition as claimed in any of claims 1 to 6, in which the alkyl group of the higher alkyl vinyl ether consists of lauryl, cetyl, stearyl or myristyl.

8. A resinous composition as claimed in any of claims 1 to 7, in which said vinyl chloride resin component consists essentially of said copolymer of a higher alkyl vinyl ether and vinyl chloride.

9. A resinous composition as claimed in any of claims 1 to 7, in which said vinyl chloride resin component consists essentially of said copolymer of a higher alkyl vinyl ether and vinyl chloride and said vinyl chloride polymer resin.

10. A resinous composition as claimed in claim 9, in which said vinyl chloride polymer resin is a homopolymer of vinyl chloride.

11. A resinous composition as claimed in any of claims 1 to 10, in which not more than 20 parts by weight of an impact modifier for vinyl chloride resins is incorporated in to 100 parts by weight of the resinous composition.

12. A resinous composition as claimed in claim 11, in which said impact modifier consists of an MBS resin, a chlorinated polyethylene, or an ethylene-vinyl acetate copolymer rubber onto which vinyl chloride has been graft polymerized.

13. A resinous composition as claimed in any of claims 1 to 12, in which at least one antimony compound is incorporated as a fire-retardant auxiliary into the resinous composition.

14. A resinous composition as claimed in any one of claims 1 to 13, in which at least one fire-retardant additive consisting of a chlorinated paraffin, hexabromobenzene, tetrabromoethane or tetrabromobisphenol A is incorporated into the resinous composition.

15. A resinous composition as claimed in Claim 1, substantially as herein described with reference to any of the specific Examples.

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