

STARCHES for EMULSIONS

as SENSORY MODIFIERS and TEXTURIZERS

PRODUCT OVERVIEW SENSORY MODIFIERS (= GRANULAR STARCHES)







PRODUCT NAME	INCI	ADDITION OF STARCH BEFORE / AFTER EMULSIFICATION	NATURALITY	ISO 16128 Ino	OIL- ABSORPTION (g Paraffin-Oil/ 100g Starch)
BOILING-RESISTANT STARCHES (="cook stable") CORN PO4 PH "B" RICE PO4 NATURAL RICE NS	distarch phosphate distarch phosphate dimethylimidazolidinone rice starch	BEFORE or AFTER BEFORE or AFTER BEFORE or AFTER	COSMOS, NaTrue COSMOS, NaTrue	0,99 0,99 0,98	34 53 46
LIPOPHILIC STARCHES AGENAFLO 9050 AGENAFLO OS 9051 AGENAFLO TS	corn starch modified aluminum starch octenylsuccinate tapioca starch, polymethylsilsesquioxane	AFTER AFTER AFTER		0,98 0,97 0,99	37 30 34
NATIVE STARCHES MAISITA 21.001 MAISITA 9040 REISITA NATURAL TAPIOCA NATURAL TAPIOCA STER BARLEY NATURAL	zea mays (corn) starch zea mays (corn) starch oryza sativa starch tapioca starch tapioca starch hordeum vulgare starch	AFTER AFTER AFTER AFTER AFTER	COSMOS COSMOS COSMOS COSMOS	1 1 1 1 1 1	35 34 49 35 36 37
ORGANIC CERTIFIED GRANULAR STARCHES MAISITA 9060 organic maize starch ORGANIC TAPIOCA NATURAL organic tapioca starch	zea mays (corn) starch tapioca starch	AFTER AFTER	ORGANIC ORGANIC	1	34 35

TEXTURIZING STARCHES

	PRODUCT NAME	INCI	NATURALITY STATUS	ISO 16128 Ino
COLD WATER SWELLABLE STARCHES	AGENAJEL 20.313	hydroxypropyl starch phosphate		0,93
	AGENAJEL 20.306	distarch phosphate	COSMOS	0,99
COOK-UP STARCHES	STÄRKINA NATURAL	solanum tuberosum starch	COSMOS	1,00
	TAPIOCA NATURAL	tapioca starch	COSMOS	1,00
	AGENAJEL 20.383	hydroxypropyl starch phosphate		0,93

BENEFITS OF STARCHES in COSMETIC EMULSIONS



SENSORY MODIFIERS

have been proven as outstanding aesthetic enhancers. Moreover, their addition to emulsion systems is a unique natural way to improve the quality of emulsions.

- Pleasant skin-feel
- Mattifying effect as the emulsion is quickly absorbed
- Easy spreading without whitening or shininess
- Reduced stickiness and greasiness

TEXTURIZING STARCHES

Starches are suitable for thickening of o/w emulsions and substitution of synthetic thickeners.

- Rheology modifying
- Thickening
- Pleasant texture

LIKELY APPLICATIONS

All cosmetic emulsions of the oil in water (o/w) and water in oil (w/o) emulsion types are suitable for optimisation with granular starches. These include products from a wide range of application areas, such as

- Suncare
- After Sun
- After Shave
- Foundations / Primer
- Daycare
- Deodorants / Antiperspirants







AGRANA'S SENSORY MODIFIERS

are green alternatives to SILICONES and mineral-oil based ingredients like NYLON/small MICROPLASTIC.

TEXTURIZERS

are natural derived alternatives to synthetic thickeners.

SUSTAINABLE PRODUCTS

- Non-GMO
- Naturally derived and safe
- Readily biodegradable
- Organic products available

GREEN ALTERNATIVES

- Gluten-free
- Exempt from REACH
- Non animal tested
- Vegan

1. PROPERTIES of GRANULAR STARCHES in EMULSIONS









TAPIOCA STARCH

RICE STARCH

PARTICLE SIZE and PARTICLE SHAPE

Depending on the raw material source (maize, rice, tapioca, potato) granular starches offer active surface area, a natural range of particle size distribution and different particle shape. Therefore starches can be a perfect alternative for small-sized NYLON powders.

PARTICLE SIZE DISTRIBUTION



Particles with a diameter of less than 40 µm are below the tactile threshold and are therefore particularly suitable as wrinkle fillers. These particles have a mattifying effect and give velvety skin feel. Potato starch granules are larger in particle size and not recommended for aesthetic modification functionality in a formulation.

1.1 QUALITATIVE OPTIMIZATION in EMULSIONS

HIGH SORPTIVE POWER

The high sorptive power facilitates the adsorption of both hydrophilic and hydrophobic liquids and the active substances dissolved in them. Consequently, substances like active ingredients, botanical extracts, vegetable oils, deodorant actives, sun filters and fragrances are also adsorbed and absorbed, providing a constant release and ensuring a long-lasting effect. REMARK: Heat sensitive actives have to be added after cooling down.

EXCELLENT MICROBIOLOGICAL and CHEMICAL PURITY

All our products are made of food-grade starches and optimized by chemical or physical treatment for cosmetic applications (see our data-sheets).

EXTENDED MOISTURISATION

As the emulsified fluid is absorbed during emulsification process it then let back down on skin.

REDUCTION OF GREASINESS

Especially in natural or suncare formulations greasy and heavy textures can be a problem when natural oils or organic UV filters are used. Granular starches reduce greasiness and too heavy textures due to the ball-bearing effect. This is a completely different mechanism for achieving a silky skin feel than for silicones (film forming) beside additional benefits of starches like moisturisation and reduction of greasiness.

1.2 SENSORIAL PROFILE oF GRANULAR STARCHES

Sensory evluation by the AGRANA sensory panel with 12 people



RESULTS: The addition of granular starches improves absorption and reduces oilness.



1.3 ADDITION of GRANULAR STARCHES

ADDITION of STARCH



ADDITION BEFORE EMULSIFICATION (= "HOT PROCESS")

only for our boiling-resistant starches like CORN PO4 PH "B", RICE PO4 NATURAL and RICE NS optimum production technique is to disperse the starch in the aqueous phase and stir until emulsification occurs. Temperatures beyond 90°C (194°F) do not influence the properties of cross-linked starches as they are resistant to boiling. The mixture should then be allowed to stand for about 15 minutes at temperatures of approx. 80°C without gelatinizing.

The subsequent processing steps is carried out using the standard procedure.

If active substances which are oily or dissolved in solvents have to be transferred to these phase depots they should be mixed with either a portion of or all the starch. After a lenght of stay of 15 minutes, if necessary under additional stirring, this pre-mix should be transferred into the corresponding emulsion phase.

ADDITION AFTER EMULSIFICATION (= "COLD PROCESS")

of all starches suitable which are not boiling-resistant can be dispersed in emulsions immediately after emulsification process – e.g. at about 45° C (113°F) – prior to commencing the cold stirring phase. This temperature should remain constant for at least 15 minutes and care should be taken that the dispersion is homogenously distributed. Optimization will depend on ensuring the temperature and stirring be maintained as long as possible.

1.4 SWELLING BEHAVIOR of BOILING-RESISTANT STARCHES

SWELLING CAPACITY OF BOILING-RESISTANT STARCHES

Boiling-resistant starches possess a notable swelling capacity but are resistant to gelatinization and do not thicken the formulation. Furthermore they can even be autoclaved without losing their spreading powers. They are stable at acidic and weakly alkaline pH-values, chemically pure, inert and compatible with standard active substances.



Absorption capacity of CORN PO4PH "B" in water at increasing temperatures.

The picture shows the results of a sedimentation test: 10 g of cross-linked starch are warmed in 100 ml water under stirring for 15 minutes at the indicated temperatures from $40^{\circ}C - 90^{\circ}C$ (104 - 194°F).

Then the suspension is filled in cylinders where the starch can sediment. This procedure demonstrates the swelling and absorption capacity of the starches during an emulsification process.

INCREASING EMULSION STABILITY

Practical experiences have shown that the use of these starches leads to a substantial increase in the stability of the emulsion ("Pickering effect"). Dehydrated and lipophilic starches contribute best to improve emulsion stability.

The dispersed starch particles do not form sediments even on protracted storage. Whilst a barely noticeable viscosity increase will occur it does not mean that the raw formulation needs changing nor does the packing opening need to be altered. Depending on base formulation with the addition of granular starch the spreading property will be improved significantly.

2. THICKENING STARCHES

2.1 PROPERTIES of COLD WATER SWELLABLE STARCHES

PRODUCT NAME	INCI	NATURALITY	DIN ISO 16128 Ino
AGENAJEL 20.313	Hydroxypropyl Starch Phosphate	_	0,93
AGENAJEL 20.306	Distarch Phosphate	COSMOS	0,99

AGENAJEL 20.313 and AGENAJEL 20.306

are cold water swellable waxy corn starch based thickeners. Both are rheology modifiers and creaminess enhancers, frequently paired with xanthan gum. They are shear-thinning with rapid viscosity recovery. Both grades leads to an opaque formulation. Usual applications as thickeners are creams & lotions, cream shampoo, masks and much more.

VISCOSITY PROPERTIES of COLD WATER SWELLABLE THICKENING STARCHES



2.2 PROPERTIES of THICKENING COOK-UP STARCHES

PRODUCT NAME	INCI	NATURALITY STATUS	GELATINIZATION TEMPERATURE
STÄRKINA NATURAL	Solanum tuberosum starch	COSMOS	~ 75°C (for 15 min)
TAPIOCA NATURAL	Tapioca starch	COSMOS	~ 80°C (for 15 min)
AGENAJEL 20.383	Hydroxypropyl Starch Phosphate	-	~ 70°C (for 15 min)

If the natural granular starches are cooked in aqueous solutions (gelatinization process), they will lose their granular structure, changed to solubilized amorphous particles and built up viscosity. At higher concentrations, the viscosity rise significantly, so that such gelatinized starches can be used to substitute synthetic thickeners in emulsions. With moderate cooking, partially gelatinized particles are obtained, which lead to a special texture and / or sensorial skin effects.

THICKENING CAPABILITY (pH related) of SWELLABLE MODIFIED STARCHES



formulations. Above pH 10 the cross-linkage of the starches breaks and so the viscosity increase, but with low stability.

2.3 PRODUCTION of **O/W EMULSIONS**

Hot versus cold process

The production of O/W emulsions is usually carried out in a hot process in which water and oil phases are heated to approx. 70°C. The emulsifying temperature depends on the melting point of the ingredients and on the critical gel network temperature of the O/W emulsifier system. Most of available emulsifiers and emollients are solid and have to be melted. This is the reason why O/W emulsion preferred produced in hot process.

Many companies want to reduce engergy costs, which is why the cold process is becoming increasingly important. Liquid emulsifieres and oils are used in the cold process. The cold water swellable starches are predestined for such a process.

- Cold water swellable starches can be used for hot and cold processes.
- Cook-up starches have to be cooked to get viscosity and are only useful for hot processes.

Incorporation of starches in Emulsions

Cold water swellable starches:

Addition in oil or water phase (single or premix; hot or cold process)

Opportunities for Incorporation of cold water swellable starches for production of O/W Emulsion

Option 1: Cold water swellable starch into oil phase

Especially if combinations of thickeners are used (for example swellable starch and Xanthan Gum), it is possible to thicken water phase with Xanthan Gum and parts of starch and rest of the cold water swellable starch in form of a premix with oils. The starch will be dispersed in oil, but not solubilized. The solubilization step of starch follows after addition into water phase and contact to water at emulsification step.

Option 2: Cold water swellable starch into water phase

The cold water swellable starch can be dispersed (single or as premix with glycerin) into the already heated water phase, or the pre-dispersed cold starch/water phase is heated to 70°C. At emulsification step, oil phase is added to water phase, to built up the emulsion.

Remark: It is important, that swellable starches are incorporated whilst intense stirring. If the formulations include oil or glycerine, it is possible to make premixes with them and add it together whilst intense stirring.



Incorporation of cook-up starches for production of O/W Emulsion

The Cook-up starch can be easily dispersed in water, whilst stirring. Afterwards the starch has to be cooked (usual ~ 70 -90°C) to obtain viscosity. At emulsification step, oil phase is added to water phase, to built up the emulsion.

Stabilization of liquid crystalline (LC) structured O/W emulsions with cold water swellable starches

It is important, that the viscosity of the water phase supports the emulsification process to produce smaller oil droplets. The viscosity can be increased by addition of swellable starch or other natural thickeners.

• The smaller the oil droplets are, the more stable the O/W emulsion is (Stokes's law).



Influence of addition of cold water swellable starches and second homogenization step

FORMULATION REFERENCE

PHASE	PRODUCT NAME	INCI	Rec. 1 % W/W	Rec. 2 % W/W	SUPPLIER
A	Supreme	Polyglyceryl-3 Rice Branate, Cetearyl Alcohol, Sucrose Stearate	5,00	5,00	Sinerga
	DUB 810C MB	Coco-Caprylate/Caprate	5,50	5,50	Dubois
	DUB MCT 5545	Caprylic Capric Triglyceride	4,00	4,00	Dubois
	Covi-ox-T9oC	Tocopherol	0,10	0,10	Kensing
В	Deionised water	Aqua	80,40	76,90	
	BLANOVA [®] Active D-Panthenol 75%	Panthenol, Aqua	0,50	0,50	Azelis
	BLANOVA® Glycerin 99,5%	Glycerin, Aqua	3,50	3,50	Azelis
С	AGENAJEL 20.313	Hydroxypropyl Starch Phosphate	0,00	3,50	AGRANA
D	Phenoxyethanol	Phenoxyethanol	1,00	1,00	
	Results viscosity	Properties			
		Flow point (mPa.s)	10.200	16.000	
		Viscosity (mPa.s)	35.000	45.000	

Manufacturing instructions

Heat phases A and B up to approx. 70°C. Add phase C to phase B and homogenize. Add phase A to phase BC and homogenize. At around 40"C homogenize again. Add phase D to phase ABC. Adjust pH to 7.

COMMENT: The formulation with AGENAJEL 20.313 (Rec 2) shows a significant increased viscosity in comparison to formulation without starch (Rec 1) - see also rheology measurement.



Stabilization Theory

According to the gel-network theory of O/W emulsions, the aqueous phase consists of a liquid crystalline (LC) gel network generated by hydrophilic emulsifiers (Hydrophilic-Lipophilic Balance = HLB > 7) and lipophilic co-emulsifier (HLB < 6) forming a layer around emulsified droplets and membranelike structures throughout the aqueous phase (Dahms 2010). The membrane-like structures are responsible for the viscosity of the emulsion as well as for the stabilization of the oil/water-interphase.

Example of an O/W Emulsion thickened with AGENAJEL 20.313. Rec 1 was formulated without starch. Rec 2 include AGENAJEL 20.313.

Rec 1 does not contain starch or any other hydrocolloid. A second homogenization step at approx. 40°C reduces the average oil droplet size. At this so-called liquid crystalline (LC) gel network temperature, the emulsifier forms multilamellar structures in the water phase. At the same time, the interfacial tension decreases significantly, and emulsion droplet sizes become even smaller.

Rec. 2 contains AGENAJEL 20.313 (Hydroxypropyl Starch Phosphate). Obviously, the presence of AGENAJEL 20.313 improves the emulsifier performance during the first emulsification step at 70°C. A second homogenization step at approx. 40°C reduces the average oil droplet size for the reasons mentioned before.

Microscopic pictures: Diluted into water; 400-fold magnification with Normarski prisma





Pictures of emulsions after 1 week at 50°C





AGENAJEL 20.313

Rec. 2b: = Rec 2a O/W Emulsion after second emulsification step at ~ 40°C

Separation stable

Rec. 1: without Starch

Rec. 2: with AGENAJEL 20.313

Regardless of the emulsion droplet size, AGENAJEL 20.313 stabilizes the emulsion by supporting the water-binding capacity of the emulsifier gel network structures and prevents the oil droplets against creaming.



COMMENT: AGENAJEL 20.313 and second homogenization step (at 40°C) improves storage stability.

3. SYNTHETIC POLYMER REPLACEMENT

Two cold water swellable and two natural granular starches were tested in comparison to synthetic thickeners in simple O/W Emulsion for their match capability of texture, flow and skin sensory aspects.

TESTED STARCHES		SYNTHETIC PRODUCTS			
Granular thickening starches Swellable thickening starches		Acrylates/C10-30 Alkyl Acrylate Crosspolymer			
TAPIOCA NATURAL AGENAJEL 20.306		Acrylates Copolymer			
STÄRKINA AGENAJEL 20.313		Carbomer			

PHASE	RAW MATERIAL	INCI	% W/W
PHASE	RAW MATERIAL	INCI	% VV/VV
A	Deionised water	Aqua	To 100,00
	Thickener		0,20 - 10,00
	Brontide	Butylene Glycol	3,00
В	NaOH 10% solution	Aqua, Sodium Hydroxide	As required
С	Syneth S10	Polyglyceryl-10 Stearate	2,50
	Syneth S8	Polyglyceryl-6 Distearate	2,50
	Lexfeel N20	Diheptyl Succinate (and) Capryloyl Glycerin/ Sebacic Acid Copolymer	10,00
D	SharoSense Plus 181-N	Maltol, Polyquaternium-80	0,70

ASSESSMENT CRITERIA

VISCOSITY (LVE, TB, 10 rpm, 30 seconds with helipath)						
RHEOLOGY MEASUREMENT						
SENSORY	SCALE 1	SCALE 5				
Pick up	No	High				
Cushioning (on application)	Light	Heavy				
Richness	Light	Rich				
Spreadability	Low	High				
Tackiness	None	Very				
Speed of absorption	Slow	Quick				
After feel	Light	Heavy				

3.1 CARBOMER REPLACEMENT

SENSORIAL AND VISCOSITY ANALYSIS



 6 sensory characteristics identical for AGENAJEL 20.306
2 sensory characteristics identical for AGENAJEL 20.313 • After feel differed by factor of 2

CONCLUSION: AGENAJEL 20.306 recommended for replacement.



• 2 differed by 1, 3 differed by 2 or more

3.2 ACRYLATES COPOLYMER REPLACEMENT

SENSORIAL AND VISCOSITY ANALYSIS



MICROSCOPIC ANALYSIS



FLOW CURVE



OVERVIEW – ACRYLATES COPOLYMER VERSUS NATURAL ALTERNATIVE

COMMENTS for TAPIOCA NATURAL
4/7 identical, 3/7 factor of 1 difference
Similar
Much higher
Very different
Smaller internal phase
Identical
Identical

CONCLUSION: TAPIOCA NATURAL is a suitable natural alternative to Acrylates Copolymer.

3.3 ACRYLATES/C10-30 ALKYL ACRYLATE CROSSPOLYMER (=AAAC) REPLACEMENT

SENSORIAL AND VISCOSITY ANALYSIS



STÄRKINA is identical on 0/7, and 7/7 differ by a factor of 1. TAPIOCA NATURAL is identical on 4/7, and 2/7 differ by a factor of 1. Viscosities are comparable.

MICROSCOPIC ANALYSIS



TEXTURAL PROFILE ANALYSIS



OVERVIEW – AAAC VERSUS NATURAL ALTERNATIVE

CRITERIA	COMMENTS FOR STÄRKINA
✓ Sensory assessment	7/7 differ by a factor of 1
✓ Viscosity	Very similar
✓ Flow point	Higher than AAAC but same shape curve
✓ Microscope	Very similar internal phase droplet size
✓ Stability	No difference
✓ Textural profile analysis	Similar profile

CONCLUSION: STÄRKINA is a suitable natural alternative for AAAC.



4. COMBINATIONS of STARCH and XANTHAN GUM for REPLACEMENT of SYNTHETIC POLYMERS

4.1 STUDY in an O/W-EMULSION

Evaluation of synergistic effect

Example of O/W Emulsion thickened by combinations of AGENAJEL 20.313 and Xanthan Gum.

The thickening efficiency of AGENAJEL 20.313 / XG combinations in comparison to synthetic thickeners (Carbomer, Acrylates / C10-30 Alkyl Acrylate Crosspolymer) was evaluated in a basic "low self-thickening" O/W Emulsion (Polygylceryl 10-Oleate was selected as emulsifier since it doesn't promote viscous-liquid crystalline structures).

G R A W	FORMUI	AT	ION	DE	TA	ILS			AGENAJEL AGENAJEL AGENAJEL Carbopol® Xanthan Go Xanthan Go	– mid amo – Iow amo um – Iow:	unt
Formulations		Form.1	Form.2	Form.3	Form.4	Form.5	Form.6	Form.7	Form.8	Form.9	Form.10
Trade names	CTFA/INCI	[w-%]	[w-%]	[w-%]	[w-%]	[w-%]	[w-%]	[w-%]	[w-%]	[w-%]	[w-%]
Phase A											
Polyaldo 10-1-0 KFG	Polyglyceryl-10 Oleate	2,00									
DUB MCT 5545	Caprylic/Capric Triglyceride	12,00									
BLANOVA [®] Tocopherol Acetate	Tocopherol	0,50									
Paratexin [®] BA	Benzyl Alcohol	1,00									
Phase B											
Deionised. water	Agua	up to 100									
Glycerin	Glycerin	4,00									
Carbopol®Ultrez 10 Polymer	Carbomer	0,20									
	Acrylates/C10-30 Alkyl										
Carbopol®Ultrez 20 Polymer	Acrylate Crosspolymer		0,20								0,20
AGENAJEL 20.313	Hydroxypropyl starch Phosphate			1,50	1,50	5,00	7,50		5,00		
Rhodicare® T	Xanthan Gum	0,10	0,10	0,10	0,50			0,50	0,10	1,00	
NaOH (18 %)	Sodium Hydroxide	0,46	0,46								0,46
	RHEOLOGY - FLOW CURVE										
	Flow point [mPas]	612	6.122	204	3.265	1.840	6.100	918	2.860	816	6.000
	Viscosity [mPas]	8.700	26.000	900	11.750	11.000	44.000	5.000	17.500	15.000	30.000
		Mid viscose	High viscose	Low viscose	Mid viscose	Mid viscose	High viscose	Mid viscose	Mid viscose	Mid viscose	High viscose

RESULTS RHEOLOGY

Viscosities (rheometer 1/sec)

The viscosities caused by the combined use of AGENAJEL 20.313 and Xanthan Gum is higher than the sum of the emulsion viscosities that can be achieved with the individual hydrocolloid amounts resulting from this blend.



This synergistic effect can be achieved even with very low levels of Xanthan Gum (0,1 %) in combination with higher amounts of AGENAJEL 20.313 up to 5 % (see right picture).





RESULT: The amplitude sweep of the O/W emulsion with 1 % Xanthan Gum shows low and G" (~ 10.000 mPa.s) which indicate low structuring components.



RESULT: The amplitude sweep of the O/W emulsion with **5 % AGENAJEL 20.313** show and G" (G'-G"= 80.000 mPa) within the linear viscoelastic range.



SUMMARY

Combinations of AGENAJEL 20.313 and Xanthan Gum can replace the synthetic thickeners in O/W lotions. The similar rheological profile of green alternatives to "Acrylates/C10-30 Alkyl Acrylate Crosspolymer" thickening systems by using the synergistic effects of starches with Xanthan Gum in O/W emulsion could be demonstrated. The 3-dimensional gel made of modified starch AGENAJEL 20.313 and Xanthan Gum is mutually stabilized with a nice creamy texture and flow curve comparable to carbomers.

Xanthan Gum concentrations should not exceed 0,2% to avoid stringy textures. Missing viscosity should be adjusted with AGENAJEL 20.313.

ıden sweep
module (storage modulus) module (loss modulus)
e flow point is ~35.000 mPa.
v G' (~ 35.000 mPa)
v G (~ 53.000 mm a)
module (storage modulus)
module (loss modulus)
s is a clear indication of much more stronger hydrogel elasticity and ucturing components induced by AGENAJEL 20.313.
e yield point is around 30.000 mPa.
rs a higher values of G' (100.000 mPa) and a bigger difference between G'
module (storage modulus) module (loss modulus)
equally high G' value (120.000 mPa) and nearly same difference between and G" (around 100.000 mPa) within the linear viscoelastic range indicates arly same strong elasticity and structuring components.
yield points of the O/W-emulsion is around 30.000 mPa.
lymer is comparable with the amplitude sweep of the O/W emulsion



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