TECHNICAL APPLICATION
INFORMATION

Herbstreith&Fox

Cloud Stabilisation of Pineapple and Passion Fruit Juice as well as Nectars produced from Concentrate

Introduction

Fruit juices are very popular with the consumer. Juices from apples as well as citrus fruits are traditional products in Europe.
With the possibility to concentrate fruit juices large progress has been made technologically; fruit juice concentrate is easier to transport as it needs less freight room.

At the same time the product becomes microbiologically more stable due to the lower aw-value in combination with the low pH-value which the product already has. These advantages made an easier import of exotic fruit products into Europe possible.

Only a few years ago pineapple juice which today is a standard product in most supermarkets was a novelty. The fruits are juice extracted in the country of origin and concentrated afterwards before they are shipped to Europe.

This lowers the production costs considerably due to the more cost-effective production and the highest production safety. In Europe the concentrate will only be diluted and then pasteurised.

However, the use of concentrate has its limits in influencing the quality of the fruit juice to be produced. The quality strongly depends on the juice production and the concentration process. The cloud stability as one of the quality criteria for the consumer may hardly or even not at all be influenced depending on the fruit. This is quite obvious in juices produced from tropical fruits. The label still says: "Shake well before use!" Rather quickly the cloud particles sediment a deposit is formed in contrast to the clear upper part.

By changing the Miscellaneous Directive the EU made it possible to improve the quality of pineapple juice and passion fruit juice as well as their nectars by adding pectin.
According to this new legal situation the industry has now the opportunity to add pectin to pineapple juice and passion fruit juice as well as their nectars in accordance with the "Fruchtsaftverordnung" (Fruit Juice Directive) resp. Directive on Fruit Nectars and Fruit Syrups.

The quantity is fixed here to a maximum of 3g non-amidated pectin per litre (E440).

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INFLUENCES
on the Cloud Stability

Cloud particles are particles which develop during compression of the fruit juice production. They are mainly cell fragments with a more or less large diameter which sediment in naturally clouded juice during storage. Stoke's Law describes the behaviour of suspended solids in an aqueous solution.

The floating behaviour of a particle expressed by the sedimentation velocity V is influenced by the particle diameter (d), the particle density (r_t), the density of the solution (r_{Fl}) and the viscosity of the solution (h); g describes the acceleration due to gravity.

$$V = \frac{d^2 (p_t - p_{FL})}{18\eta} g$$

According to this law simplified applicable for the problems of the cloud stabilisation,

1. the diameter of the cloud particles,
2. the difference of the density of the cloud particles and the surrounding solution and
3. the viscosity of the surrounding solution

influence the floating behaviour of the cloud particles in the fruit juice.

The following measures improve the cloud stabilisation of fruit juice:

1. increase of the fine cloud percentage by technologically useful measures (for example homogenisation),
2. decrease of the density difference of the cloud particles and the surrounding solution,
3. increase of the viscosity of the surrounding solution.

The cloud particles of pineapple juice have been examined in various pieces of work. Here the cloud had been differentiated into coarse cloud with an average particle size of 100 µm and fine cloud with 0.5 µm.

According to Stoke's Law coarse cloud cannot be kept floating due to the large particle diameter. It would have to be removed by separators for producing a cloud stable juice. Sensorically this juice cannot be labelled naturally clouded juice because of the low cloud content (Will, 1995).

In contrast apple juice can be produced cloud stable with the application of suitable processes. The juice contains a large amount of fine cloud and pectin so that throughout the storage time only little cloud will be deposited.

The fine cloud shows a lesser intention to sediment due to the smaller particle size and the dissolved pectins increase the viscosity of the juice.

But the composition of the cloud particles have also an influence on the cloud stability of the fruit juice.

The cloud consists inter alia of proteins, lipids, neutral polysaccharides, pectin and other substances like minerals (Peceroni, S.; Gierschner, K.; 1993, Dietrich, H.; Gierschner, K.; Peceroni, S.; Zimmer, E.; Will, F.; 1996). The literature discusses different models for building a stable cloud particle. The cloud particels contain a core consisting inter alia of protein which is positively charged. This positively charged core is able to build a complex with negatively charged pectin.

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Through the strong water binding characteristics of the hydrocolloids a hydrate shell forms around the cloud particle so that the density of the cloud particle is adjusted to the serum. Furthermore the formation of a protein-polyphenol complex is discussed (Peceroni, S.; Gierschner, K.; 1993). These speculations are supported by those of Yamasaki et al. (1964) in which cloud particles contain a positively charged protein core surrounded by a carbohydrate shell consisting among others of a negatively charged pectin.

Pineapple juice has a low hydrocolloid content of only 1800 mg/l (Will, F. et al., 1994).
According to Will, F. et al. (1994) the chemical composition of the pineapple colloids possibly influences the cloud stability. Next to proteins they mainly consist of polysaccharides with an uncommonly high part of mannose the degree of galacturonic acid in pineapple juice is comparably low.
In addition the pineapple juice has a larger content of coarse cloud with a particle size of approx. 100 µm (0.9 to 4.7 g/l) whereas the fine cloud is only 0.1 - 0.9 g/l (Will, 1995). Both factors – the coarse cloud and the low pectin content – negatively influence the cloud stability.

By adding pectin the viscosity of the surrounding medium can be increased which has a positive effect on the cloud stability.
According to Will, F. et al. (1999) a very large amount of Pectin of 3 g/l had to be added to the pineapple juice produced from fresh fruits in order to label the product cloud stable.
However, due to its mushy overall impression the product received negative marks for sensory parameters.
For the production of a cloud stable juice a large quantity of pectin had to be added in order to keep the relatively large cloud particles floating by a sufficiently high viscosity of the serum.

A homogenisation step in which the size of the cloud particles was reduced was an improvement resulting in a slower sedimentation speed of the cloud particles.
During production of the pineapple juice additional interactions between the newly developed cloud particles and the added pectin molecules may build as synergistic effects which may lead to protein-pectin-complexes making the production of a cloud stable juice only possible with a combination of homogenisation and pectin addition.

In trials of the University of Hohenheim pineapple juice had been produced from concentrate stabilising the cloud by adding pectin. These trials proved that the particle size moved towards a smaller size if a homogenisation step took place after dilution of the juice.
Furthermore it was noted after examination of the cloud particles that the pectin had formed a bond with the cloud particles. Without the addition of pectin the particles were positively charged whereas the particles were negatively charged after the addition of pectin (Mensah-Wilson, M. et al.; 2000).

The aim of the here described trials was to enhance the cloud stabilisation of juices from different fruits by combining the addition of pectin with homogenisation by receiving optimal sensory parameters.
Pineapple juice produced from fruit juice concentrate was hereby in the centre of attention. Next, passion fruit nectar was produced and examined.

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Influences of Pectin *on the Sedimentation Behaviour of the Produced Goods*

Testing

Pineapple juice was produced by diluting the pineapple juice with water and adding Pectin Classic AJ 201 resp. Pectin Instant CJ 204. Furthermore a passion fruit nectar was produced.

A centrifugal test which simulates the life of the product for 1 year with the influence of normal gravity (Dietrich, H. et al.; 1996) characterised the cloud stabilising effect of the pectin application. The pectins Classic AJ 201 as well as Instant CJ 204 are pectins which have been developed at Herbstreith & Fox for their application in fruit juice drinks for the increase of viscosity. Their field of application is mainly in the development of an improved mouth-feel in low-calorie fruit juice drinks.
For this, pectins will be standardised for developing a constant viscosity. Pectin Instant CJ 204 is an agglomerated citrus pectin which disperses directly in the fruit juice whereas Pectin Classic AJ 201 disperses in the fruit juice concentrate to create optimal conditions for the dissolving behaviour.

In these tests both pectins had been used in a dosage of 0 - 0.24%. The pectins had been dispersed in the juice concentrate and the necessary amount of water for diluting the concentrate was added.

The mixed concentrate was then heated to 95°C. A part of it had been homogenised with a pressure of 100 bar at this temperature. Afterwards the juice was bottled and cooled at room temperature.

After two days the samples were analysed to show the influence of the pectin dosage and the homogenisation.

The determination of the cloud stability was made according to the above mentioned centrifugal test. For this, the juice was centrifuged at 4200 g for 15 minutes and the cloud of the serum was determined before and after the centrifuging with a cloud photometer. With this centrifuge test the life of a sample which stands upright under the influence of normal gravity is simulated for 1 year.

The cloud of the serum, in the following called stable cloud, was referred to the measured cloud of the juice sample before centrifugation and is called relative turbidity.

According to Will, F. (1995) a pineapple juice may be called absolutely cloud stable if a relative turbidity of 40% is reached.

$$\text{Relative Turbidity [\%]} = \frac{T_{Ü}\ [FNU]}{T_S\ [FNU]} \cdot 100$$

$T_{Ü}$: Turbidity of supernatant after centrifugation
T_S: Turbidity of the juice

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At the same time the stable cloud (cloud of the supernatant after centrifugation) should be at least 300 FNU. This value may be used as a limit under which the juice will be seen as being cloudy by the human eye. If these parameters are met the juice can be called a cloudy juice after an appropriate long storage time.

Furthermore the viscosity of the supernatant had been measured after centrifugation (serum viscosity) with a rotation viscosimeter (coneplate measuring system) at a defined shear rate (D = 50 1/s).

Cloud Stabilisation of Pineapple Juice

Influence of the Homogenisation Step on the Cloud Stability

The influence of the homogenisation on the relative cloud is shown in fig. 1. The untreated control as well as the sample which has only been homogenised with 100 bar show the lowest values of the relative turbidity (5.9% resp. 4.6%). Juice to which 0.08% pectin has been added during production showed with 11% a significantly higher, yet not high enough value. The combination of homogenisation and 0.08% Pectin Classic AJ 201 showed the best results (relative turbidity: 43%).

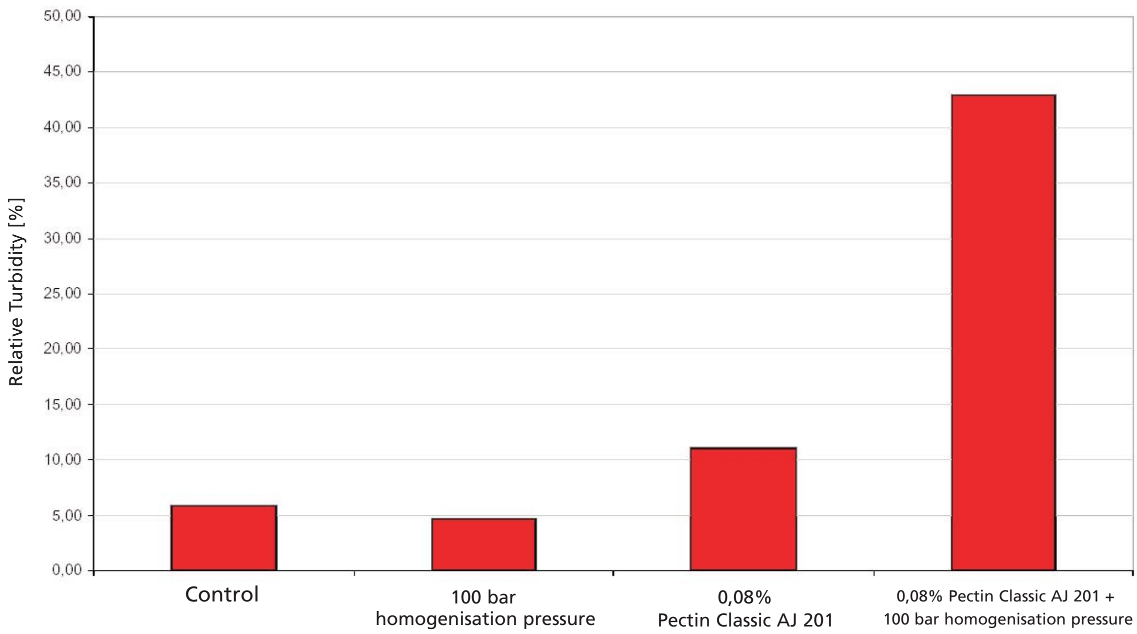

Fig. 1: Sedimentation behaviour of pineapple juice under the influence of Pectin Classic AJ 201 (0.08%) and homogenisation (100 bar)

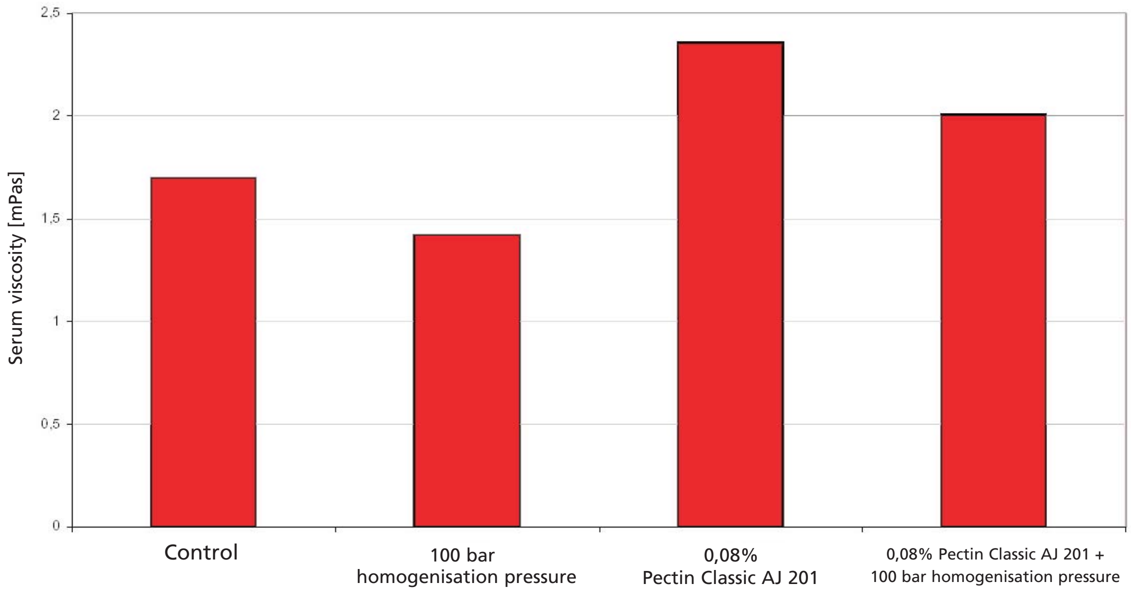

Fig. 2: Serum viscosity of pineapple juice at 20° C under the influence of Pectin Classic AJ 201 (0.08%) and homogenisation (100 bar)

	Initial Cloud [FNU]	Stable Cloud [FNU]
Control	1341	79
Homogenisation with 100 bar	1626	76
Addition of 0.08% Pectin Classic AJ 201	1476	164
Addition of 0.08% Pectin Classic AJ 201 and homogenisation with 100 bar	1566	673

Table 1: Initial cloud and stable cloud of pineapple juices

Table 1 shows that the stable cloud will increase with pectin, yet the stable cloud will only be achieved in combination of addition of pectin and homogenisation.

This explains the different influences on the floating behaviour of the cloud particles. Large particles will be crushed due to homogenisation which increases the initial cloud.

Because of the smaller particle diameter the sinking speed will be lower.

Yet, this effect will not suffice to produce a stable pineapple juice. Only the addition of pectin which will result in an increase of serum viscosity (see fig. 2) and stabilisation of the cloud particles will improve the cloud stability noticeably.

Concentration of Pectin Classic AJ 201 and Pectin Instant CJ 204 Influences Cloud Stability

Fig. 3 shows the sedimentation behaviour of the produced pineapple juice under the influence of pectin concentration of Classic AJ 201 resp. Instant CJ 204. The sedimentation depends on the dosage of the respective pectin. Without the addition of pectin the juice was not cloud stable, the value of the relative turbidity was 6%. The sedimentation beha-viour was noticeably improved by adding Pectin Classic AJ 201 resp. Instant CJ 204.

From a dosage of 0.08% Pectin Classic AJ 201 a cloud stable juice had been produced by using the tested concentrate. The value of the relative turbidity was larger than 40%. The stable cloud also meets the requirements being larger than 300 FNU (see table 2).

To be able to produce a cloud stable pineapple juice 0.16% Pectin Instant CJ 204 was necessary. The reason for the higher dosage of Pectin Instant CJ 204 is the different standardisation of the two pectins.

The serum viscosity increased steadily depending on the pectin dosage (see fig. 4). Adding Pectin Classic AJ 201 had a stronger influence on the serum viscosity than using Pectin Instant CJ 204. The higher serum viscosity – achieved by addition of pectin – had a positive effect on the sedimentation behaviour. The pectin addition influenced the cloud stability, however, only to a certain concentration. The relative cloud approached a constant value. From a dosage of more than 0.12% pectin the effects of pectin on the cloud stability in this series of experiments was hardly noticeable.

A further increase of the pectin concentration lead to an increased viscosity and thus to an increased mouth-feel of the juice.

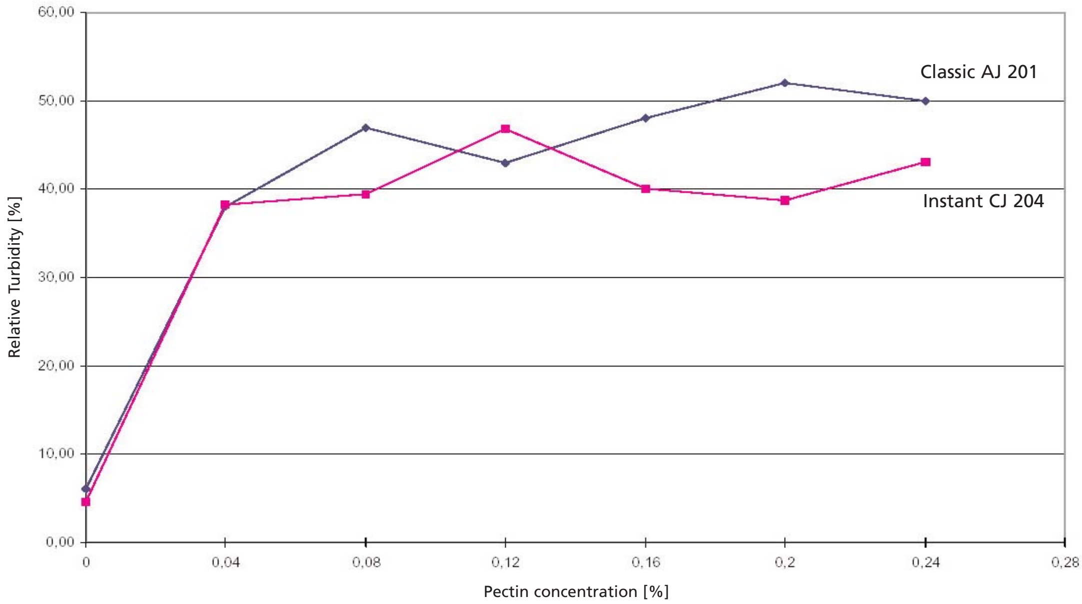

Fig. 3: Sedimentation behaviour of pineapple juice influenced by Pectin Classic AJ 201 resp. Pectin Instant CJ 204

	Stable Cloud [FNU]						
Pectin dosage [%]	0.00	0.04	0.08	0.12	0.16	0.20	0.24
Pectin Classic AJ 201	91.4	792	704	932	778	1078	924
Pectin Instant CJ 204	68.5	573	591	703	600	581	646

Table 2: Stable Cloud [FNU] depending on pectin dosage

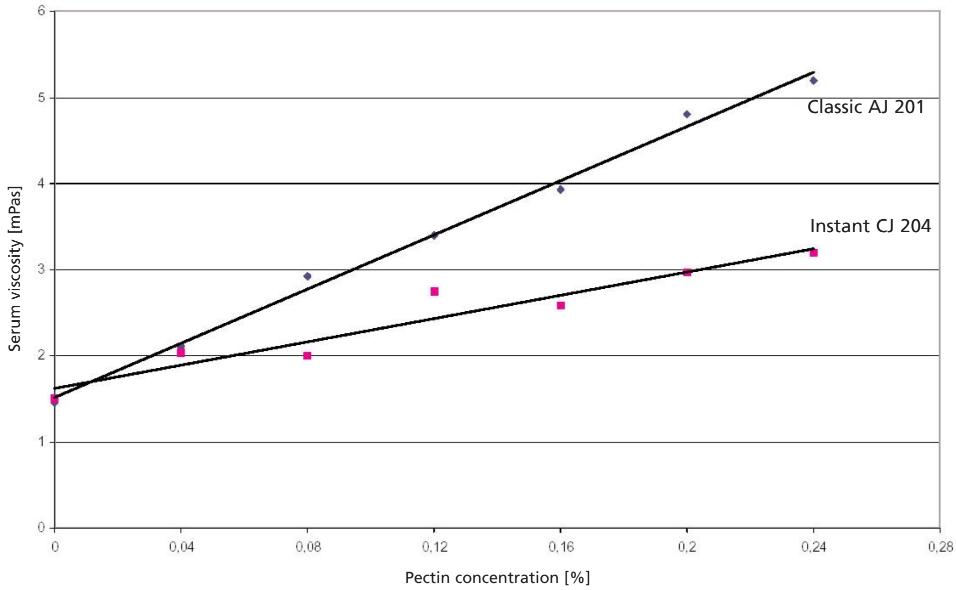

Fig. 4: Serum viscosity of pineapple juice at 20°C in dependence from concentration of Pectin Classic AJ 201 resp. Pectin Instant CJ 204

Cloud Stabilisation of Passion Fruit Nectar

According to the procedure accomplished in the previous chapter also passion fruit nectar was produced from concentrate and stabilised by adding pectin and homogenisation. For these experiments a nectar with 25% fruit content was produced according to official regulations.

By adding Pectin Classic AJ 201 combined with homogenisation the cloud stability of the passion fruit nectar was greatly improved.

Already the addition of 0.04% Pectin Classic AJ 201 resulted in a more stable product with a relative turbidity of 37%.

The addition of Pectin Classic AJ 201 results in a constant increase of viscosity. As in the experiments for the stabilisation of pineapple juice the curve of the relative turbidity approached a constant value with an increasing pectin dosage. From a dosage of 0.2% of Pectin Classic AJ 201 no substantial improvement of the sedimentation behaviour is noted.

Sensorically the sample which is not stable had been judged watery. By adding pectin the mouth-feel of the nectar was increased and it gained a more full-bodied flavour.
The stable samples with the addition of 0.08% and 0.12% Pectin Classic AJ 201 had been judged as very good. The addition of 0.16% pectin resulted in an exceptionally strong mouthfeel which leads to a stronger impression of sweetness of the product.

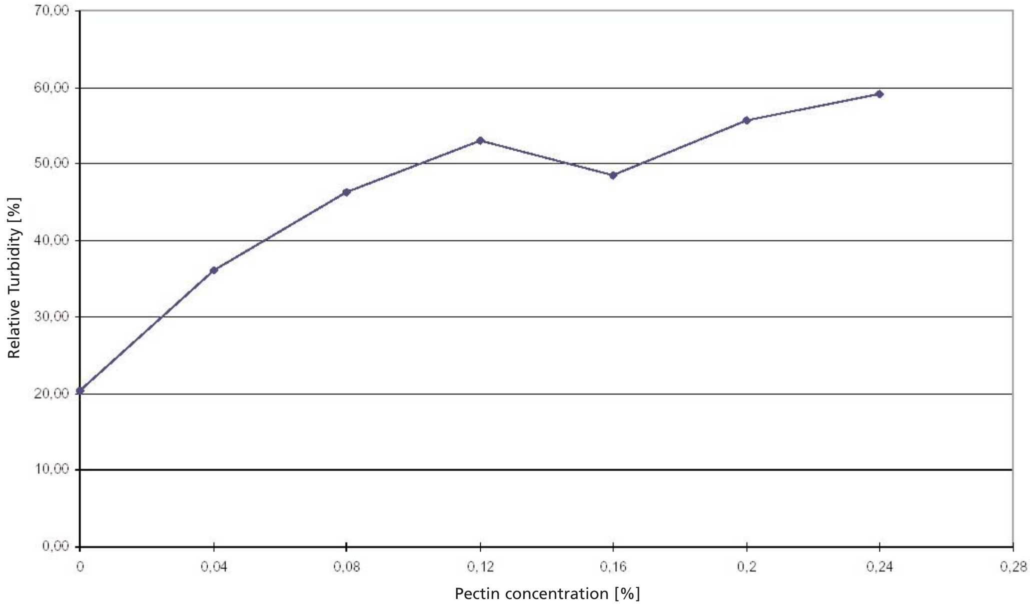

Fig. 5: Sedimentation behaviour of passion fruit nectar influenced by Pectin Classic AJ 201

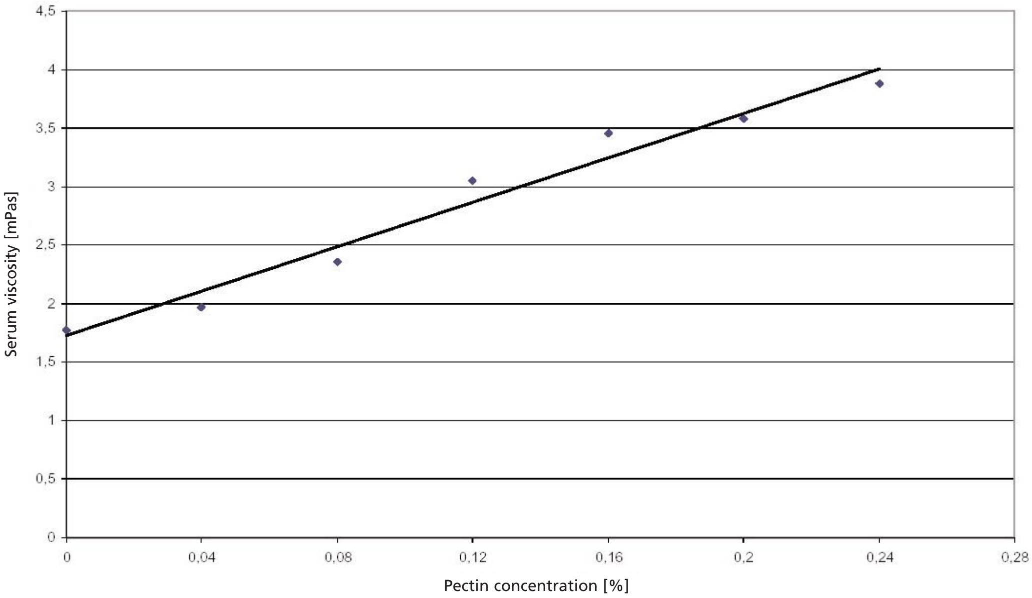

Fig. 6: Serum viscosity of the passion fruit nectar at 20° C in dependence from concentration of Pectin Classic AJ 201

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The cloud stability of a juice depends among others on the size of the cloud particles. According to Stoke's equation the diameter of the cloud particles influences the sedimentation speed in a stronger way than the viscosity of the serum.

Therefore coarse cloud can only be limitedly stabilised by the influence of viscosity. Pineapple juice produced in a series of experiments by Will, F. et al. (1999) contained a too small part of fine cloud for gaining a cloud stable product. By crushing the coarse cloud in the production of pineapple juice the share of fine cloud, whose sedimentation speed is lower, increases.

The addition of pectin results in an increase of viscosity of the serum which also has a positive effect on the sedimentation behaviour.

Yet only the combination of an increase of fine cloud by homogenisation and pectin addition gives the opportunity for producing a cloud stable pineapple juice. With a dosage of 0.08% Pectin Classic AJ 201 a pineapple juice can be produced which shows a relative turbidity of > 40% and a stable cloud of > 300 FNU. This juice can be called cloud stable.

Because of the described experiments the process for the production of pineapple juice resp. passion fruit juice must include two steps:

1. addition of pectin
2. homogenisation of the juice

Pectin can be dispersed directly in the fruit juice concentrate. Then further ingredients will be added. After mixing the ingredients the juice will be homogenised (homogenisation pressure for example 100 bar).

The high shear rate which effects the product crushes the coarse cloud.
At the same time the added pectin dissolves and can sediment on the newly formed cloud particles. Then flavour concentrate is added and the juice de-aerated. Before bottling the juice resp. nectar will be pasteurised.

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Summary

The addition of high-methoxyl Classic pectins improves pineapple juice and passion fruit nectar produced by diluting the concentrate, in an important criteria: The cloud stability.

Even a small pectin dosage of Pectin Classic AJ 201 resp. Instant CJ 204 resulted in an improved sedimentation behaviour.

The cloud stability, expressed in the relative turbidity, had been improved so far that the juice showed a very low sedimentation after a storage time of 1 year.
This was achieved with a dosage of 0.08% Pectin Classic AJ 201 resp. 0.16% Pectin Instant CJ 204. Depending on the used pectin type the cloud stability reached a value which was not much further influenced by an additional increase of the pectin dosage.

The positive effect on the cloud stability was attributed to the increase of the serum viscosity in the juice.

A possible complexing of positive protein particles of the cloud particle with the added negatively charged pectin is being discussed as another possibility. This reaction leads to a hydratised hydrocolloid shell which adjusts the density of the cloud particle to the density of the serum. This reaction stops as soon as the proteins are saturated with pectin. A further increase of the pectin dosage had no influence on the cloud stability.

A homogenisation step is necessary during the production process to crush the coarse cloud of the juice and to increase the fine cloud. These two measures make it possible to keep the pectin dosage small for stabilisation and thus to increase the viscosity of the juices and nectars as little as possible. The sensory analysis of the so stabilised juices did not show any loss of "juiciness".

A change of regulation for the production of fruit juices from pineapple concentrate and passion fruit concentrate put the fruit juice industry in a position to improve the quality of their pineapple and passion fruit juices and nectars by adding Classic Pectin. Thus the consumer wishes for a cloud stable product have been fulfilled.

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