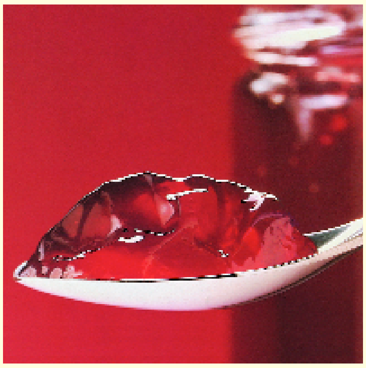

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# Chances and Limits for the Use of Pectin as Emulsifier

*November 1998, Lecture of the Master Class on Emulsion Technology held at FI Food Ingredients Europe, 3-5 November 1998, Frankfurt, Germany*

# CHANCES AND LIMITS
## *for the Use of Pectin as Emulsifier*

**Introduction**

Pectins are mainly used as gelling or thickening agents in the food industry but also in the non-food industry. The application of pectins as emulsifier and emulsion stabilizer is yet not known in much extend.
In the following the emulsifying and emulsion stabilizing properties and the utilization of these properties in practical applications have been investigated.
The possibilities but also the limits for the use of pectin as emulsifier will be shown.

**1 Pectins**

Pectin is a natural substance and as a component of the middle lamella and the primary cell wall an important structural element of all fruits and vegetables. It acts as a bonding substance with supporting and stabilizing functions and controls due to its great swelling ability and hydrocolloid nature the plant's water system.

**1.1 Structure**

Pectin is mainly formed with D-galacturonic acid molecules which are linked to each other by a -(1,4)-glycosidic bonds to become polygalacturonic acid. The carboxyl groups are partially esterified with methanol. In some cases the secondary alcohol groups of the pectins can also carry acetyl groups. In beet pectins also ferolyl groups could be found (see fig. 1).

The presence of neutral sugars such as galactose, arabinose or xylose which are linked as side chains to the pectin macromolecule and the rupture of the main chain caused by rhamnose make pectin to a branched zig-zag-shaped heteropolysaccharide.
In the plant's cell the pectin molecules are so tightly bound to other molecules of the cell wall that they cannot be extracted with water. This water insoluble form is called protopectin.
During the ripening of fruit or the boiling of vegetables protopectin is converted into soluble pectin.

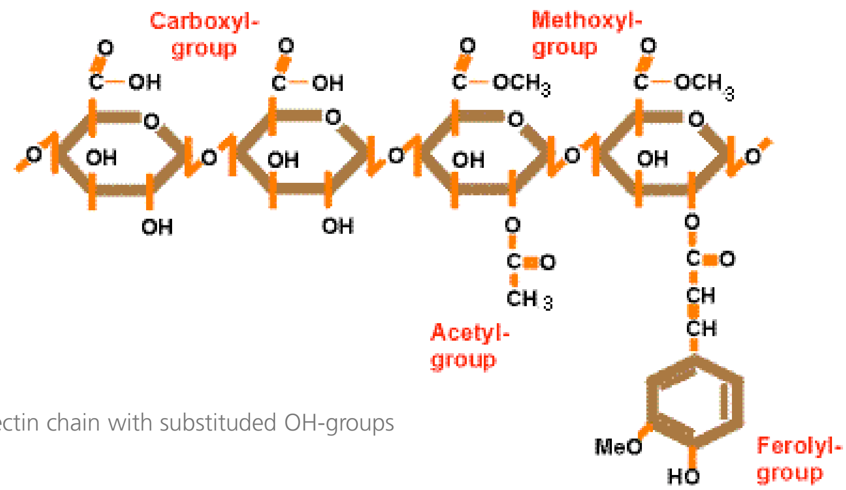


Fig. 1: Pectin chain with substituded OH-groups



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### 1.2 Raw material and pectin production

All vegetable raw materials with a high pectin content are suitable for the production of pectins. For the industrial production the prime sources are apple pomace, citrus peels and sugar beet chips. The various raw materials yield different amounts of extractable pectin.

| | |
|---|---|
| Apple pomace: | 10-15% |
| Sugar beet chips: | 10-20% |
| Sunflower-infructescence: | 15-25% |
| Citrus peels: | 20-35% |

Pectins extracted from various raw materials can be different in molecular structure (i.e. molecular weight, degree of esterification, acetyl content, neutral sugar content, distribution of the methoxylated carboxyl groups) and therefore possess different functional properties.
Pectins in which the degree of esterification of the galacturonic acid residues is > 50% are known as high methoxyl pectins. They require the presence of a low pH-value and a high concentration of sucrose for gelation. Such conditions help to minimize electrostatic repulsions between polymerchains. Polymer-solvent interactions lead to the formation of a gel network.

Low methoxyl pectins with degree of esterification < 50% gel in the presence of calcium ions.

The extraction of soluble pectin in industrial scale is carried out by means of acid hydrolysis resulting in liquid pectin. This liquid pectin can be dried to pectin extracts or the pectin is isolated from its aqueous solution by alcohol precipitation. Afterwards it is dried, grinded and sieved to a defined particle size. Pectins as well as pectin extracts are used in this study.

## 2 Emulsions

Emulsions are disperse multiphase systems containing at least two immiscible liquid phases (disperse phase and continuous phase). Depending on the type of disperse phase there are two emulsion types: water in oil (w/o) and oil in water (o/w) (see fig. 2) (Stang, Schubert, 1996). Typical o/w food emulsions are milk, mayonnaise, dressings and various beverages. Butter and margarine are examples of w/o emulsions.

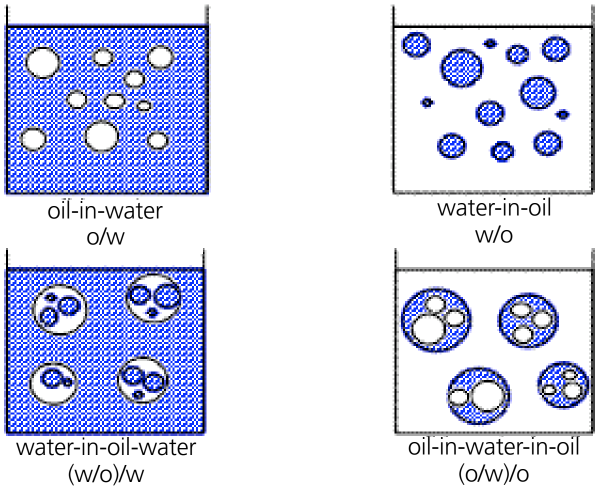


Fig. 2: Different types of emulsions



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### 2.1 Quality of food emulsions

**The essential characteristics decisive for quality of food emulsions are shown in figure 3 .**

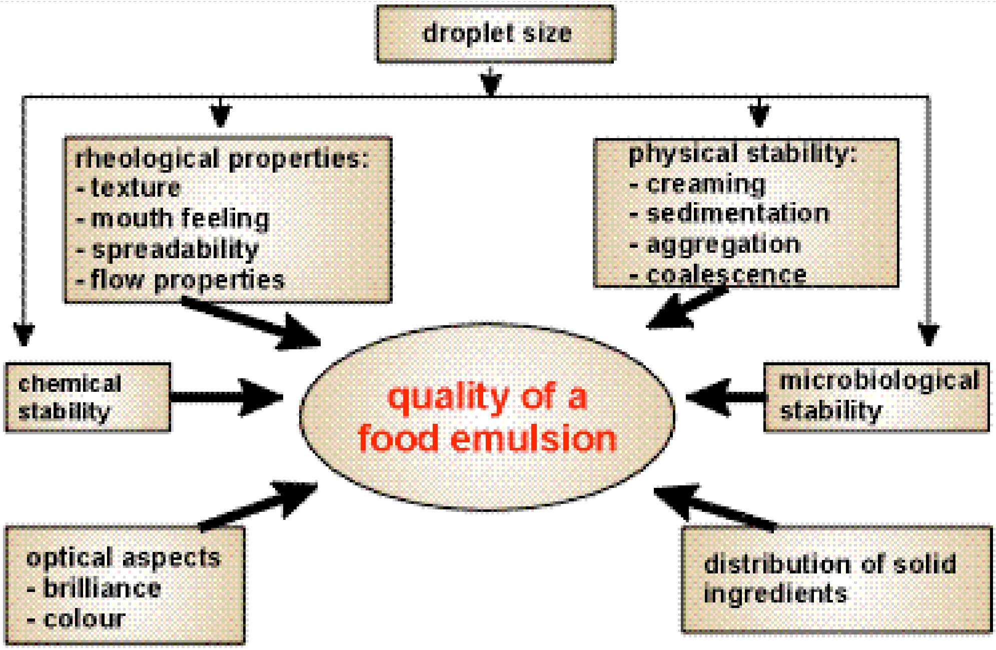


Fig. 3: Quality of food emulsions (Stang, Schubert, 1996)

**Very important factors for the quality of emulsions are droplet size and droplet size distribution. The latter influences physical stability, rh e ological properties and optical aspects of an emulsion. The quality of emulsions is often checked by measuring the droplet size besides rheological properties.**

**The droplets in food emulsions are widely dispersed in size and may range from 0.1µm to 10µm. The smaller the droplet size of the disperse phase the more stable is the emulsion from the physical point of view. The size of the droplets to be achieved in the emulsion process is depending on the adsorption kinetic of the emulsifier and the choice of a suitable emulsifying equipment. The adsorption kinetics describe how fast newly formed interfaces are covered with emulsifier molecules.**

### 2.2 Stability

**Emulsions are thermodynamically instable. The system tends to reduce the interface between lipophilic and hydrophilic phase by coalescence. This can lead to a complete phase separation.**

**Basically three mechanisms can cause physical instability of emulsions (see fig. 4):**



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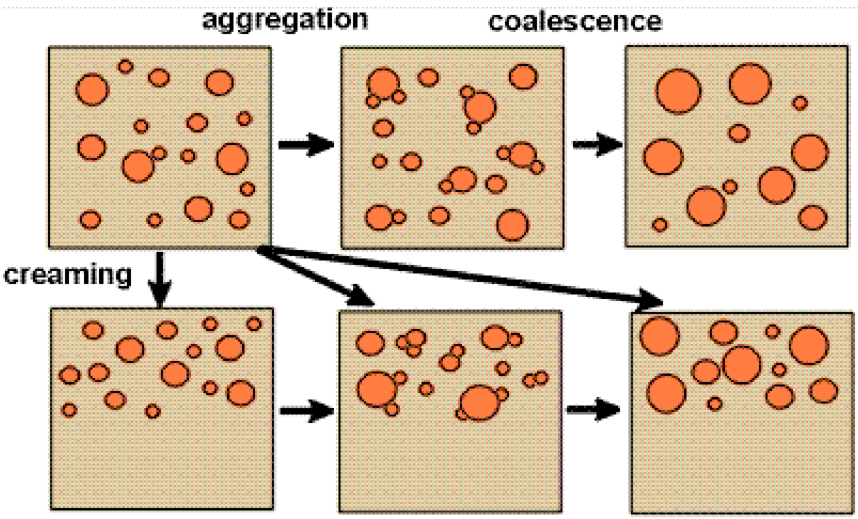


Fig. 4: Factors of physical instability of an o / w Emulsion (Stang, Schubert, 1995)

**Creaming of oil droplets in an o/w emulsion caused by density differences between both phases. The droplet size distribution is changing only locally.**
**Due to attraction forces between the droplets they can aggregate. Within the aggregates the d roplets remain separated by a thin film consisting of continuous phase. The droplet size distribution of droplets forming aggregates remains constant. The initial droplet size distribution can be regained by shaking or stirring.**
**Droplet coalescence however causes a real change in droplet size distribution. The initial droplet size can be regained only by another emulsifying process.**

### 2.3 Emulsifier and stabilizer

**To improve the stability of emulsions emulsifying agents divided in emulsifiers and stabilizers are added. The significance of emulsifiers and stabilizers as well as the factors influencing the physical stability of emulsions can be demonstrated by Stokes' law:**

$$VSt = \frac{D\ r \bullet g \bullet x2}{18 \bullet h\ c}$$

**VSt = stokes sinking (or creaming) velocity**
**D r = density differences between the phases of the emulsion**
**g = acceleration, due to gravity**
**x = droplet diameter**
**h c = dynamic viscosity of the continuous phase**

**The physical stability of an emulsion can be improved according to Stokes' law by increasing the viscosity of the continuous phase by adding stabilizers or reducing and maintaining the droplet size. In some cases sticky emulsions also have a certain yield point caused by a hydrocolloidal stabilizer preventing floating of the oil droplet.**
**Stabilizers (thickening agents) are dissolved in the continuous phase and increase its viscosity or create a yield point. This restricts the moveability of the droplets and with that less collisions between the droplets occur reducing also the risk of coalescence.**
**Typical stabilizers are polysaccharides.**



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Emulsifier molecules (surfactants) have hydrophilic and hydrophobic parts. They adsorb at the interface between the disperse and the continuous phase thus forming a dense film around the droplets of the disperse phase. This film prevents the coalescence of the droplets.

## 3 Pectins as emulsifiers and emulsion stabilizers

Hydrocolloids function as emulsion stabilizer by thickening and increasing the viscosity of the aqueous phase so that the tendency of the dispersed oil globules to migrate and coalesce is inhibited or minimized.
Some gums such as gum arabic can also stabilize emulsions by means of a protective colloid. The gum is adsorbed at the interface to form a coating around the dispersed globules or particles, thus imparting a charged surface to the coated particles which then repel each other and maintain a stable, dispersed state.
Also some hydrocolloids can perform as surfactants and reduce surface tension to allow easier formation and maintenance of stable emulsions.
The hydrocolloid pectin has been widely used as gelling and thickening agent in the food industry for many years. As mentioned before the prime source of industrial pectins are apple pomace, peels of citrus fruits and sugar beet chips.
The pectin which can be extracted from sugar beet differs from that of apples and citrus fruits. In addition to galacturonic acid, neutral sugars and methoxyl substituents sugar beet pectin also contains considerable quantities of acetyl groups.
The presence of the acetyl groups should enhance the hydrophobic nature of the molecule, giving it a surface active character with potential as an interfacial agent in oil / water emulsions and air / water foams.

Depolymerized citrus and apple pectins are also said to possess excellent emulsifying and stabilizing properties; but this thesis could not be confirmed in this study (UK 2 311 024).
The emulsifying properties have been investigated in this study by determination of the surface tension of apple, citrus and beet pectin solutions.
The stability of o/w emulsions prepared with different pectin types of different sources (apple, citrus, beet) have been investigated by measurement of the change of oil droplet size distribution during storage of the emulsions.
As a practical application pectins were also tested for the stabilization of citrus oil emulsions. Beet and citrus pectins were compared with gum arabic.

## 4 Use of pectin as emulsifier

### 4.1 Determination of the surface tension of pectin solutions

The adsorption kinetics of an emulsifier describes its time dependant adsorption at the interface between two fluid phases, that means how fast newly formed interfaces are covered with emulsifier molecules. The adsorption kinetics of emulsifiers is most frequently measured in terms of surface tension as a function of surface age called the dynamic surface tension.

The dynamic surface tension was measured with the drop volume tensiometer Lauda TVT1. The principle of the drop volume method consists in exact determination of the volume of a drop which detaches from a capillary (see fig. 5). By increasing the volume of a drop its weight is increased until it reaches a critical value at which it cannot be counterbalanced by the surface tension and the drop detaches. The critical drop volume is proportional to the surface tension.



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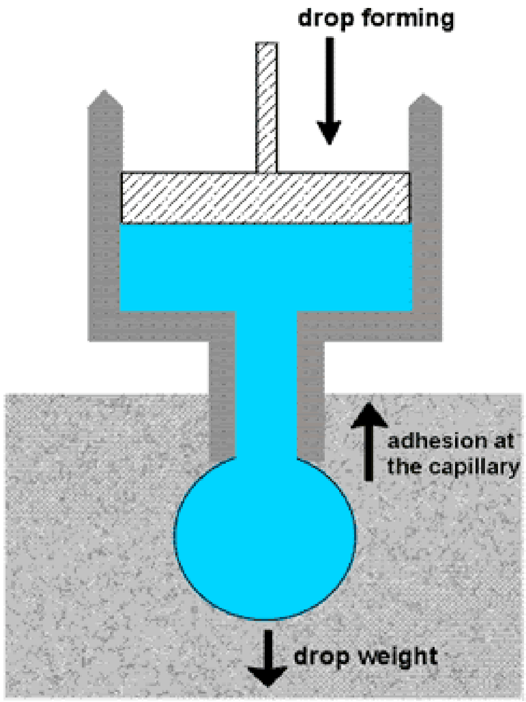


Fig. 5: Drop-volume-method to determine the dynamic s u rface tension

The dynamic surface tension was determined from aqueous solutions of high methoxyl apple and citrus pectin, beet pectin (dosage 2.5%), drum dried apple and beet pectin extract (dosage 10%).
The results are shown in figure 6.

Beet pectin and beet pectin extract show the most significant decrease in surface tension. This can be explained by the presence of acetyl groups (4 - 5 %) in the beet pectin molecule which enhances the hydrophobic nature and thus the surface active character.
The significant decrease in surface tension of the pectin extracts especially the beet pectin extract cannot be explained by the pectin content in these extracts. Other substances present in the extract must have an influence on the surface tension, too.
In spite of these measurements the results of the stability test of the o/w emulsions prepared with pectin extracts were negative (see 5.2).

## 5 Use of pectins as emulsion stabilizers

### 5.1 Preparation of o/w emulsions with different pectins

o/w emulsions have been prepared with pectins manufactured from different raw materials (apple, citrus, beet), with different degree of esterification (HM, MM, LM), with amidated pectins, dried pectin extracts, depolymerized apple and citrus pectins (see table 1).

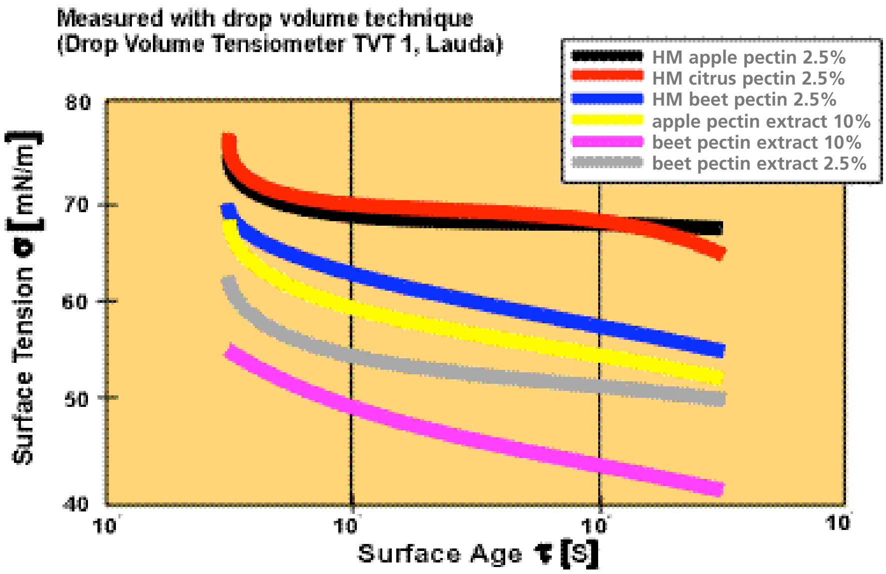


Fig. 6: Dynamic surface tension of different pectin solutions



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| | | Degree of esterification [%] | Viscosity 2.5% solution D = 250 $s^{-1}$ [mPas] |
|---|---|---|---|
| 1. | High methoxyl apple pectin | 70 | 400 |
| 2. | High methoxyl apple pectin | 60 | 780 |
| 3. | Medium methoxyl apple pectin | 50 | 600 |
| 4. | Low methoxyl apple pectin | 38 | 600 |
| 5. | Amidated apple pectin | 30/19* | 150 |
| 6. | Medium viscous apple pectin | 60 | 30 |
| 7. | Low viscous apple pectin | 62 | 5 |
| 8. | High methoxyl citrus pectin | 70 | 350 |
| 9. | High methoxyl citrus pectin | 60 | 200 |
| 10. | Medium methoxyl citrus pectin | 50 | 210 |
| 11. | Low methoxyl citrus pectin | 38 | 900 |
| 12. | Apple pectin extract (25 % pectin) | 65 | |
| 13. | Beet pectin extract (35 % pectin) | 60 | |

* Degree of amidation (%)

Table 1: Pectins used for the preparation of o/w emulsion

| | | Degree of esterification [%] | Galacturonic acid [%] | Molecular weight | Acetic acid [%] | N-content [%] |
|---|---|---|---|---|---|---|
| 14. | Beet pectin I | 57 | 66 | n.d.** | 3.3 | 0.57 |
| 15. | Beet pectin II | 59 | 66 | 30000 | 4.6 | 0.14 |
| 16. | Beet pectin III | 56 | 69 | 30000 | 4.8 | 0.66 |

** not determine

Table 2: Recipe and preparation of the o/w emulsions

**Preparation of the o/w Emulsions**

33.0% Plant Oil

64.5% Water

2.5% Pectin

**Recipe 2**

33.0% Plant Oil

57.0% Water

10.0% Pectin Extract

**Realization**

Pectin respectively pectin extract was disloved in hot (85°C) water (Ultra Turrax).
Oil and cold pectin solution was mixed for 1 minute (Ultra Turrax 13500 min) and homogenized for 2 minutes (Ultra Turrax 24000 min).

### 5.2 Stability of o/w emulsions

The stability of the o/w emulsions was determined by measurement of the oil droplet size with laser difraction (Malvern Mastersizer X) shortly after preparation of the emulsions and after storage for 3 weeks at 50°C. In addition the viscosity of the emulsions was measured (see table 3).

| | | Viscosity D = 100 $s^{-1}$ [Pas] | Sauter Mean (after prep.) [μm] | Sauter Mean (21 days, 50°C) [μm] | Sensoric impression (21 days, 50°C) |
|---|---|---|---|---|---|
| 1. | HM-A 70 | 3.3 | 6.3 | 6.5 | stable |
| 2. | HM-A 60 | 6.1 | 5.5 | 5.7 | stable |
| 3. | MM-A 50 | 5.3 | 7.7 | 10.8 | seperated |
| 4. | LM-A 38 | 4.7 | 8.9 | 10.5 | seperated |
| 5. | A-A 30/19 | 1.6 | 14.4 | 37.6 | seperated |
| 6. | M-Visc A | 0.3 | 4.0 | 4.9 | seperated |
| 7. | L-Visc A | 0.1 | 83.5 | 101.5 | seperated |
| 8. | HM-C 70 | 3.8 | 6.0 | 5.8 | very stable |
| 9. | HM-C 60 | 1.9 | 5.9 | 5.6 | seperated |
| 10. | MM-C 50 | 1.8 | 10.5 | 9.8 | seperated |
| 11. | LM-C 38 | 1.2 | 12.5 | 40.3 | gel |
| 12. | APE | 1.8 | 33.4 | 44.6 | seperated |
| 13. | BPE | 1.7 | 11.5 | 21.1 | seperated |
| 14. | HM-B-I | 1.6 | 4.7 | 4.6 | less stable |
| 15. | HM-B-II | 0.2 | 4.5 | 4.3 | less stable |
| 16. | HM-B-III | 0.2 | 4.3 | 4.3 | less stable |

| | | | | | | |
|---|---|---|---|---|---|---|
| HM- | high methoxyl | M-Visc | | medium viscous | -A | apple |
| MM- | medium methoxyl | L-Visc | | low viscous | -C | citrus |
| LM- | low methoxyl | | | | -B | beet |
| APE | apple pectin extract | | | | | |
| BPE | beet pectin extract | | | | numbers: | DE/DA |

Table 3: Determination of oil droplet size and viscosity of o/w emulsions prepared with pectins



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### 5.3 Results

There was no increase in droplet size of the emulsions prepared with high methoxyl apple and citrus pectins if the viscosity of the emulsion was significantly increased. The sensory perception of the emulsions showed stable products. In this case no aggregation or coalescence has been occured.

The high methoxyl pectins cause an increase of the viscosity of the continuous, the aqueous phase of the emulsions. In this case high methoxyl pectins act as emulsion stabilizers, additionally the hydrophobic nature of the methoxyl groups can be discussed as emulsifying property.

Emulsions prepared with the beet pectin showed smaller droplet sizes and also no increase in droplet size during storage. The stabilizing effect of beet pectin may be due to its surface active property (see 1.1). Beet pectin reduces the surface tension and this facilitates the disruption of the oil droplets and induces repulsive forces between the droplets and prevents coalescence and creaming. Beet pectins are – because of their low molecular weight – low viscous and therefore they do not provide a significant contribution to the stabilization of the emulsion by increasing the viscosity. Beet pectins seem to act as emulsifier.

Some of the pectin emulsions show constant droplet size during storage but the emulsions were instable. This can be explained by a forming of aggregation of the oil droplets followed by creaming (see 2.2).

Low methoxyl pectins, amidated pectins and depolymerized pectins did not give satisfying results in stabilizing these kind of emulsions. To some extend the emulsions separate shortly after preparation.

## 6 Use of pectins in citrus oil emulsions

### 6.1 Determination of the stability of citrus oil emulsions

For the assessment of the emulsifying and stabilizing properties of pectins in a practical application citrus oil emulsions were selected.

Citrus oil emulsions are used as cloudy emulsions, as opacity builders for citrus beverages, the so-called soft drinks. These emulsions are often stabilized with gum arabic. Citrus oil emulsions contain mostly 10% of citrus oil and are used in the soft drink in a 1:100 dilution. Gum arabic is added at high levels up to 20% to an aqueous sugar solution and emulsified with an oil phase consisting of citrus oil and possibly weighing agents.

Beet pectins from different production charges and citrus pectins have been applied to stabilize citrus oil emulsions.

**Preparation of Citrus Oil Emulsions**

| | |
|---|---|
| 10 parts | Citrus Oil (weighed) |
| 90 parts | Water |
| 5 parts | Pectin |

**Recipe 2**

| | |
|---|---|
| 10 parts | Citrus Oil (weighed) |
| 90 parts | Water |
| 20 parts | Gum Arabic |

**Realization**

Pectin, respectively gum arabic was dissolved in hot (85°C) water (Ultra Turrax).

Oil and cold stabilizer solution was mixed (pre-emulsion). The pre-emulsion was homogenized (Microfluidizer).



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Citrus Oil (density 0.842 g/ml) was weighed with glycerolester of wood resin (E445) to a final density of 0.968 g/ml.

Pectin, respectively gum arabic was dissolved in hot water. Citrus oil was mixed to the cold solution or the preparation of a pre-emulsion. The pre-emulsion was homogenized with a Microfluidizer (see fig. 7). This homogenizer type works as opposed jet dispersing system. Two jets of the preemulsion collide and the oil droplets are disrupted. The homogenizing pressure can be changed by volume flux (Stang, Schröder, Schubert, 1998).
The citrus oil emulsions have been homogenized at different pressure levels in the range of 50 - 1000 bar.

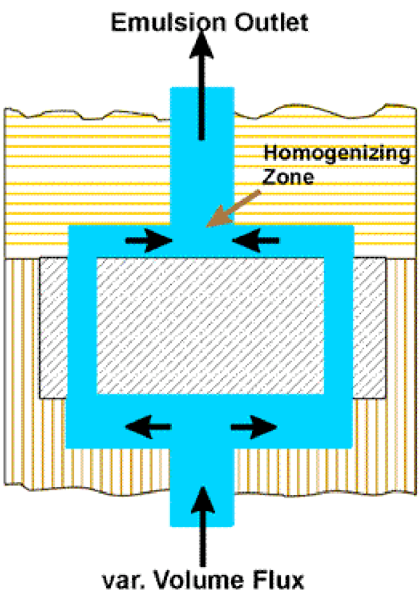


Fig.7: **Microfluidizer**

The oil droplet size has been determined by laser difraction (Malvern Mastersizer X) shortly after the preparation of the emulsions and after storage for 2 weeks at 50°C. The stability of an aqueous dilution (1:100) was also determined.

The sauter means x3.2 of the obtained droplets as a function of the homogenizing pressure are shown in figure 8.

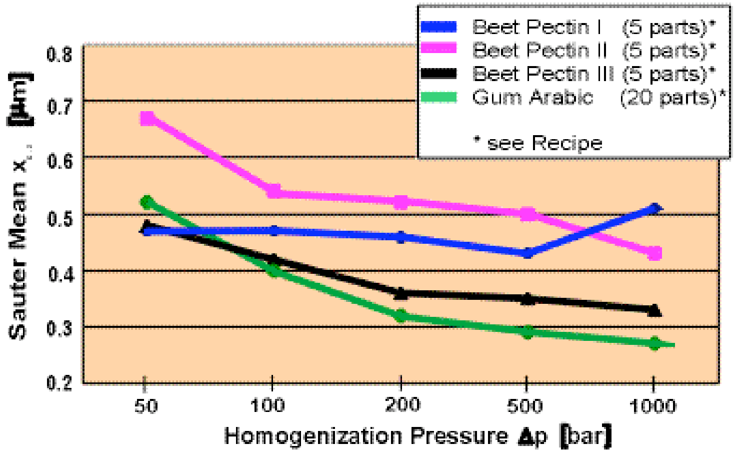


Fig.8: Dependence of Droplet Size from Homogenization Pressure

The oil droplet size of the emulsion emulsified with beet pectin II and gum arabic as a function of homogenizing pressure are comparable but the pectin dosage (abt. 5%) is much lower than the dosage of gum arabic (abt. 20%).

The oil globule size of the citrus oil emulsions is less than 1 µm, a range where stable emulsions can be expected.

Figure 9 shows the distribution of the oil droplet size of a diluted citrus oil emulsion after storage (2 weeks at 50°C) prepared with pectin and gum arabic.



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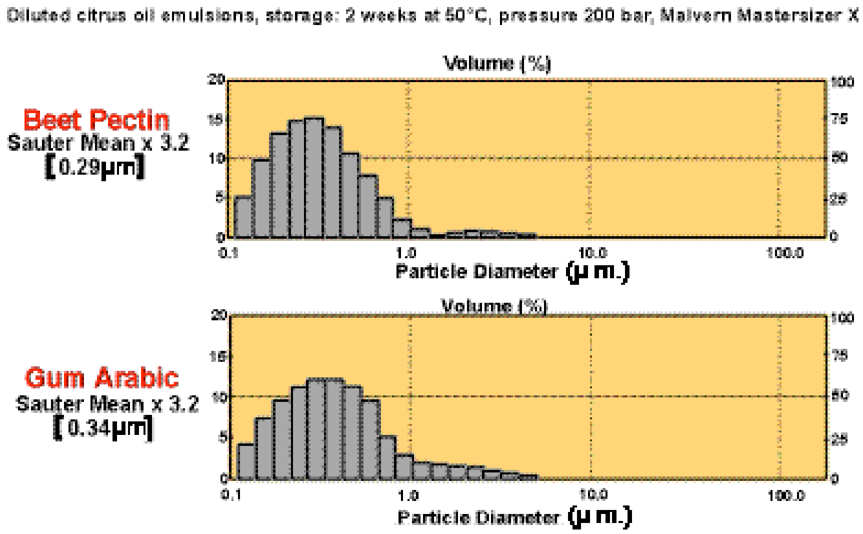


Fig. 9: Distribution of Particle Size

**In figure 10 citrus oil emulsions prepared with a citrus pectin and a beet pectin are compared. particle size of the citrus pectin emulsion (5 µm) indicates an instability. The high viscous pectins seem to be unsuitable for this application.**

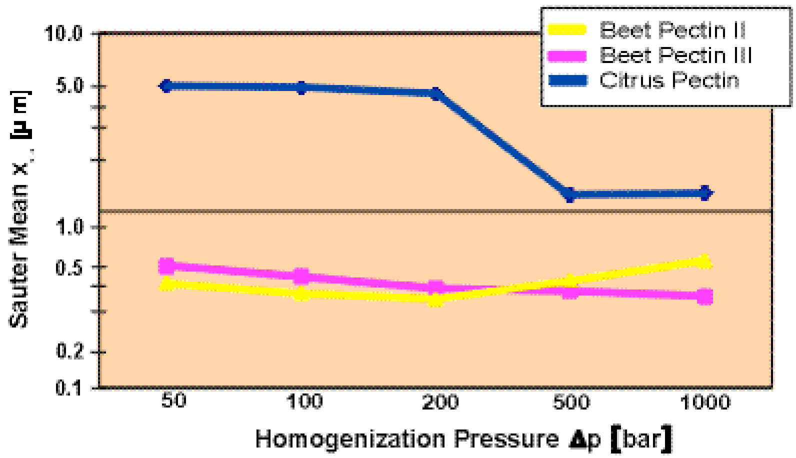


Fig. 10: Emulsifying Trials with Pectins



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### 6.2 Stability of citrus oil emulsions during storage

Fig. 11 shows the oil droplet size of citrus oil emulsions immediately after preparation and after storage for 2 weeks at 50°C. The oil droplet size of the non diluted emulsion was increasing duringstorage.

But the sauter mean x3.2 of the oil droplet distribution was still far below 1 µm.

The diluted (1:100) citrus emulsion did not show an obvious change in droplet size during measurement. No coalescence could be observed, the emulsions were stable.

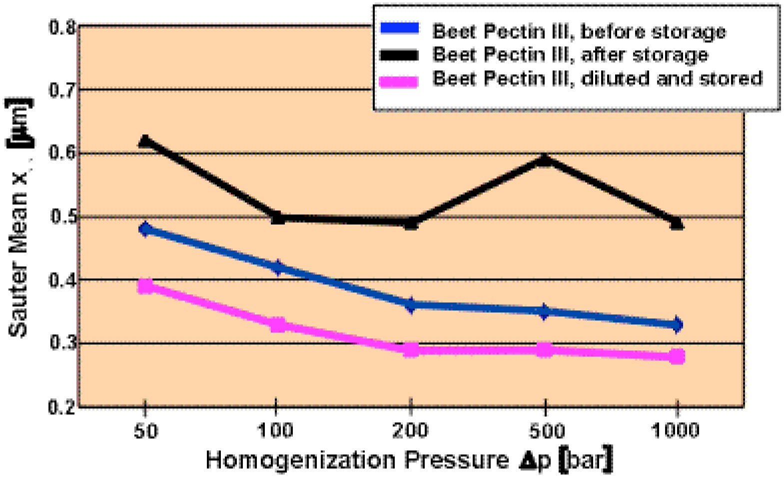


Fig. 11: Storage stability of "pectin stabilized" citrus oil emulsions

### Conclusion

Certain pectins possess emulsifying and / or emulsion stabilizing properties. The emulsion stabilizing properties of apple and citrus pectins are mainly based on the increase of the viscosity of the aqueous phase of an emulsion. The movement of the oil droplets is hindered and therefore the tendency of coalescence or aggregation of the droplets is reduced.

For the application as an emulsion stabilizer high viscous, high methoxyl pectins are suitable. Usually high methoxyl pectins show a high viscosity. In our trials the best stabilization of an o/w emulsion was obtained with a high methoxyl citrus pectin but also the high methoxyl apple pectins gave products with acceptable stability.

Possible applications for the pectins as emulsion stabilizer are mayonnaise products, dressings but also cosmetic products. In these applications high methoxyl citrus and apple pectins can completely or partially substitute other hydrocolloids or emulsifiers.

Beet pectins are due to their low viscosity not so suitable for the stabilization of o/w emulsions with 33 % oil. The surface activity of beet pectin is not high enough to emulsify the o/w emulsions.

The emulsifying properties of pectins are mainly based on the presence of hydrophobic groups to give the molecule a surface active character. Acetyl groups enhance the hydrophobic nature. In our tests the beet pectins with their high content of acetyl groups show the highest decrease in surface tension. With these pectins good results in stabilizing citrus oil emulsions could be obtained. In this application the effectiveness of beet pectins is superior to gum arabic. The pectin dosage for reaching the same stabilization is much lower than the gum arabic dosage.

In this study it could be shown that it is right to judge defined pectins as emulsifiers with an effectiveness comparable or superior to other hydrocolloids. The emulsifying properties of these hydrocolloids cannot be compared with high effective emulsifiers. Nevertheless these pectins and especially the beet pectins possess a great potential for natural products.

The results of this study reveal new and selective applications for pectins as stabilizers and emulsifiers. Further research work has to be done to learn more about the emulsifying properties of pectins and to obtain optimized food formulations.

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Research & Development
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