

## TEXTURAL AND SENSORY PROPERTIES OF MILK PROTEINS GELS MADE BY FERROUS SALTS

**Zardasht K. Ali**

Assistant Lecturer

Food Science and Quality Control  
Department, College of Agricultural  
Sciences Engineering, University of  
Sulaimani, Iraq  
[zardasht.ali@univsul.edu.iq](mailto:zardasht.ali@univsul.edu.iq)

**Jasim M. S. Al-Saadi**

Professor

Dairy Science and Technology  
Department,  
College of Food Sciences, AL-Qasim  
Green University, Iraq  
[jasim\\_salih@fosci.uoqasim.edu.iq](mailto:jasim_salih@fosci.uoqasim.edu.iq)

### ABSTRACT

The textural and sensory properties of a ferrous-induced milk protein gel prepared by heating skim milk with 12–27 mM added ferrous chloride and ferrous sulphate were investigated. Hardness and WHC increased and SWS decreased with increasing concentration for non-flavoured gel, while hardness and WHC of the flavoured gel increased and SWS decreased during storage at 7 °C for 28 days. Sensory studies showed that the gels were acceptable at added ferrous concentrations of 15–21 mM, but 18 mM got the highest score. Adding sugar and flavours did not affect the formation of the gels but increased their sensory scores.

**Keywords:** Milk gel, Ferrous, Sensory, Texture

### INTRODUCTION:

Milk protein gelation is an important process in the production of dairy products. It happens when interactions between protein and protein lead to the creation of a three-dimensional network composed of molecules of water. It is essential to balance the appealing forces needed to create a network with the repulsive forces needed to avoid its collapse<sup>(13) (19)</sup>.

Heating milk at 70 °C with added ion salt at 20 to 200 mM concentrations causes the proteins to be coagulated with whey release<sup>(16)</sup>. However, a gel is formed without whey release when milk is heated with lower concentrations of added an ion chloride<sup>(15)</sup>. Gels can also be created by altering colloidal relationships with varying pH, salt, ionic strength, and enzyme action<sup>(7)</sup>. It has been studied the thermal stability of skim milk with an ion salt added up to 20 mM by determining the time profile of pH–heat coagulation<sup>(18)</sup>, also recently showed that

heating milk with 7–20 mM has added an ion salt to form a gel similar to that of yoghurt but pH is near to natural pH of milk<sup>(17)</sup>.

Some variables influence the formation and strength of dairy gels, but the concentration of electrolytes such as calcium and iron salts is one of the most significant. The connection between ionic strength and gel formation was also proved<sup>(6)</sup>. This paper investigated the textural and sensory properties of milk gels produced by iron salts.

### MATERIALS AND METHODS:

#### *Milk samples:*

The raw milk obtained from 10 cows from the field of the college. Skim milk was produced by centrifugation (2500 g at 5 °C for 30 min).

#### *Preparation of milk gel by ferrous salts:*

Skim milk samples (100 mL) were heated for 20 min at 90 °C, cooled to ~ 22 °C, adding 18 mM from both ferrous chloride and ferrous sulfate. The milk heated again to 90 °C for 20 minutes and left for 12 hrs at least to produce a gel. All tests were done in triplicates.

#### *Gel hardness:*

A Texture Analyzer (TA) was used to determine the gel hardness at  $20 \pm 2$  °C. The parameters of the tool were: cylindrical probe; the velocity of penetration 1.0 mm /s; the distance of penetration 10 mm with 5.0g trigger <sup>(3)</sup>. The strength of gels was measured in triplicate and expressed as g/cm<sup>2</sup>.

#### *Spontaneous whey separation:*

Determination of spontaneous whey separation had been done by taking a cup of milk gel has been taken from the fridge at  $7 \pm 1$  °C and whey removed from the sample surface by a needle attached to a syringe and the cup of milk gel was weighed again <sup>(1)</sup>. To prevent further leakage of whey from the curd, the method lasted less than 10sec.

#### *Water holding capacity (WHC):*

Water-holding capacity (WHC) of gels has been determined <sup>(8)</sup>. Briefly, 10 gels were centrifuged at 5000xg for 10 min at 5 °C and calculated depending on the following formula:

**WHC % =  $[1 - (w2 / w1)] \times 100$**   
[w1: the weight of gel used, and w2: the weight of whey after centrifugation].

#### *Sensory evaluation:*

The samples assessed by 8 qualified panellists staff from Food Science and Quality Control dept. , Agricultural Science

Engineering College, Sulaimani University. The gels evaluated for flavour, texture, body, bitterness, appearance and colour <sup>(5)</sup>.

#### *Statistical Analysis:*

All data were analyzed using Statistical Program (XL stat, 2016). The significant differences between means of traits included in this study were determined using the probability ( $p \leq 0.05$ ).

## **RESULTS**

### **Effect different concentration on texture and sensory properties**

#### *Effect of ions salts concentration on gel strength, water holding capacity and Spontaneous whey separation:*

Hardness is the force necessary to attending deformation in gel, its common measure to indicate the strength of a gel network. Ions salts were not added at levels higher than 27 mM as these cause the gel strength to increase so much that coagulation occurs with the expulsion of whey <sup>(16)</sup>.

The hardness of gel prepared ferrous salts after adding 12 mM were (28, 20) but increased till reached (52.5, 50) at concentration 27 mM for fecl2 and feso4 respectively.

These results indicate that gel hardness increased with increasing concentration used for the preparation of the gels; this increase may be related to the effect of ion in increasing the number and strength of bonds between milk proteins <sup>(2) (9) (10)</sup>.

It has shown in the below table (Table 1), there were significantly different between all used concentration (12, 15, 18,21, 24 and 27mM) for both ferrous chloride and sulphate.

Table 1: Hardness (gm/cm<sup>2</sup>), Water holding capacity (%) and Spontaneous whey separation (ml) of non-flavoured gel at different concentration

Concentration (mM)	Hardness		WHC		SWS	
	FeCl <sub>2</sub>	FeSO <sub>4</sub>	FeCl <sub>2</sub>	FeSO <sub>4</sub>	FeCl <sub>2</sub>	FeSO <sub>4</sub>
12	28	20	29.22	26.45	7.00	7.60
15	39	31	23.93	32.77	5.20	6.30
18	44.5	38.5	21.53	25.03	3.60	3.30
21	36.5	62.5	22.23	26.75	3.10	2.50
24	64.5	43.5	21.84	23.40	2.40	2.00
27	52.5	50	24.33	23.06	1.70	1.80
<b>LSD (P≤0.05)</b>	<b>0.89</b>		<b>3.663</b>		<b>0.385</b>	

Water holding capacity means the capacity of gel to hold water, and with the increment of WHC, the quantity of water in the gel will be increased, while spontaneous whey separation is measuring the volume of whey separation on the top of the gels sample.

Table 1 showed also the WHC of manufactured gels by using ferrous salts. These values were changed with the concentration until reached the lowest value at the highest concentration (27 mM) for ferrous sulphate, but for ferrous chloride lowest value detected in 18 mM concentration.

WHC decreased with increasing concentration, and maybe due to the linkage of ions with milk protein in each concentration. SWS decreased with increased ions concentration (Table 1), this is due to ions enhancing aggregation of the milk proteins and reducing the pore size of the protein network, thus reducing the separation of whey from the gel <sup>(11) (12)</sup>.

#### *Sensory evaluation of non-flavoured gels:*

Sensory evaluation scores of non-flavoured ions salt milk gels are summarized in (Figure 1). In a preliminary study, added ions salts concentrations of 3–30 mM were used. Concentrations <12 mM did not result in a gel-like texture and concentrations >27

mM gave a distinct bitter taste and too much hard texture and not much bitterness detected in gels containing 12–27 mM added ferrous salts, so depend on that chosen these concentrations.

As shown in Figure 1, the total score of sensory evaluation of milk gel made by ferrous chloride and sulphate at different concentration. It notices that at first and at 12 mM concentration, got a high score because taste and bitterness cannot detect but score decrease at 15 mM and then got the highest score at 18 mM concentration because had good body, texture and appearance beside colour which are main parameters to decide.

#### **Textural and sensory properties of flavoured and sweetened ions salt milk gels:**

Because gels made with 18 mM added ferrous salts which gave the best sensory score in non-flavoured gels, this level was used to prepare flavoured sweetened gels. The added sugar and flavourings did not adversely affect the manufacture of the gels.

#### *The gel hardness of Ions-induced milk gels*

Gel strength increased significantly with the number of ions added and time of storage, till 2<sup>nd</sup> weeks (Table 2). This increase is

related to the increase in the number and strength of cross-linking of the milk proteins by ions<sup>(10)</sup>.

There was no significant difference between (0) week and first week, but there were significantly different between 2<sup>nd</sup> week which had the highest hardness with all rest for both ferrous chloride and sulphate (Table

2), maybe that related to web induced result of crosslinking.

We must notice that sugar decreases the gel strength because it limits the water availability for casein micelle hydration which is necessary for the formation of a strong gel<sup>(4)</sup>.

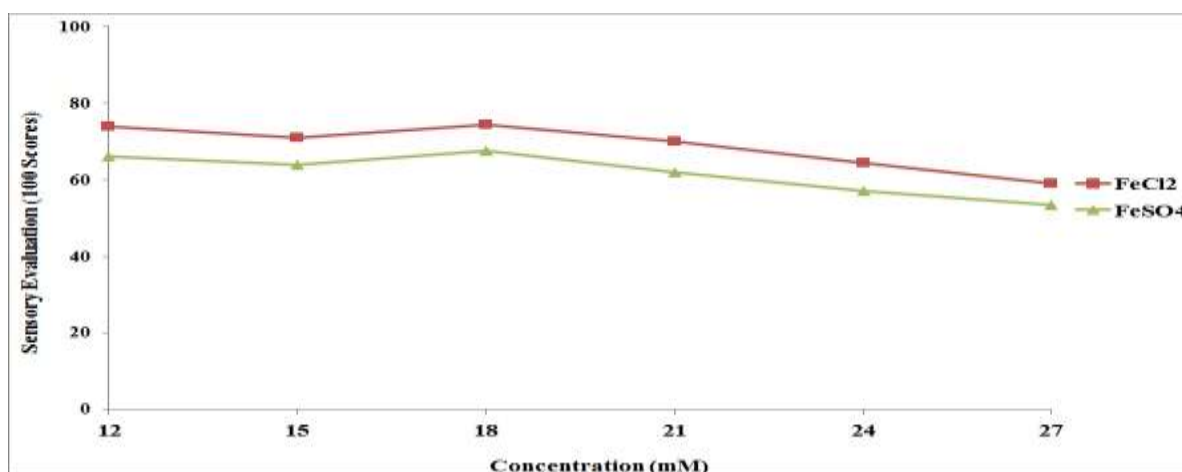


Figure 1: Sensory evaluation of non-flavoured milk gel prepared by using ferrous chloride and sulphate.

Table 2: Hardness (gm/cm<sup>2</sup>) for flavoured gels of ferrous salts during storage at 7±1 °C for up 28 days.

Flavours	Week	Ferrous Chloride	Ferrous Sulphate
Melon	0	28.5	33
	1	29.5	33.5
	2	41	39.5
	3	40.5	32
	4	37.5	39
Mint	0	31.5	31.5
	1	32	31.5
	2	39.5	42.5
	3	34.5	35
	4	41.5	38.5
Cacao	0	32.5	26
	1	33	26
	2	35.0	30
	3	23.5	30.5
	4	36.5	29.5
LSD(P≤0.05)		3.237	1.585

*Water holding capacity and spontaneous whey separation of flavoured gel during storage time*

Water holding capacity increased with increased ions concentration and time of storage (Table 3). This is due to ions enhancing aggregation of the milk proteins and reducing the pore size of the protein network, thus reducing the separation of whey from the gel <sup>(11) (12)</sup>.

It notices water holding capacity changed during storage time, which increased till 2<sup>nd</sup> and 3rd week for gels made by ferrous salts

in all flavours but after that decreased, except in ferrous sulphate increased again after that decreasing.

Generally there was significant difference between 2<sup>nd</sup> and 3th weeks with rest, and its show to be related to increasing cross-linking between ions and casein which casein to whey protein ratio decreases, the network becomes finner, cross-links become denser, and the pore sizes smaller, leading to increase of WHC <sup>(1) (14)</sup>, also there were significant difference between different flavoured used because nature of each flavour and its amount used.

Table 3: Water holding capacity (%) and spontaneous whey separation (ml) for flavoured gels of ferrous salts during storage at 7±1 °C for up 28 days.

Flavour	Week	WHC		SWS	
		Ferrous Chloride	Ferrous Sulphate	Ferrous Chloride	Ferrous Sulphate
Melon	0	32.80	41.15	0.57	0.38
	1	33.19	41.79	0.58	0.40
	2	58.30	44.71	0.56	0.60
	3	41.83	49.04	0.54	1.57
	4	42.94	51.48	1.10	0.33
Mint	0	31.55	42.03	0.75	0.73
	1	31.87	42.32	0.77	0.75
	2	53.99	45.62	0.65	0.50
	3	46.35	43.05	0.93	0.72
	4	45.10	52.58	0.32	0.24
Cacao	0	39.87	42.84	0.65	0.41
	1	40.01	43.04	0.68	0.43
	2	51.81	53.62	1.20	0.22
	3	40.85	48.87	1.14	1.62
	4	50.44	50.66	0.60	0.75
LSD(P≤0.05)		2.83	2.02	0.18	0.24

Also in the same table (Table 3), showed a significant difference between different flavours used for preparation milk gels by ferrous chloride and sulphate during storage time. At first of storage, spontaneous whey separation is normal and decrease with the time passing resulting network made by cross-linking became stronger so amount of separated water became less, but bypassing storage time that network became weak so separation water became more <sup>(1) (14)</sup>, and that is reason why amount of spontaneous whey separation increased.

#### *Sensory evaluation of flavoured gels*

Overall, the flavoured gels had very high acceptability, higher than the unflavored gels (Figure 1). With increasing sugar content, the sensory scores for flavour, texture, bitterness and appearance/colour increased for all gels.

During storage for 28 d, the total scores of the flavoured gels prepared ferrous salts decreased, while the total scores for the gels prepared with different flavours changed slightly within passing storage time. The highest sensory score, 88 out of 100, for the gel prepared by ferrous chloride and ferrous sulphate with cacao flavour, indicates high acceptance for this product. Among the sensory evaluation scores, flavour and bitterness were the most important.

Generally, there was no significant difference between zero time with the first week and first week with the second week, but clearly can see obvious difference between the other weeks, especially between first week and last week (4<sup>th</sup>), resulting of gel strength became less, and analysis protein made bitterness increased and got less degree as shown in Table 4, 5.

Table 4: Sensory evaluation for flavoured gels prepared by using ferrous chloride during storage at 7±1°C for up 28 days.

Flavour	Week	Flavour 40	Bitterness 20	Body 15	Texture 15	Appearance & colour 10	Total 100
<b>FC Melon</b>	0	34	17.5	14	14	8	<b>87.5</b>
	1	33	17.5	14	14	8	<b>86.5</b>
	2	31.5	16	13.5	14	8	<b>83</b>
	3	28	14.5	13	13.5	7.5	<b>76.5</b>
	4	25	12.5	13	13	7.5	<b>71</b>
<b>Mint</b>	0	33.5	17	14	13.5	8	<b>86</b>
	1	33	17	14	13.5	8	<b>85.5</b>
	2	31.5	16	13.5	13.5	8	<b>82.5</b>
	3	29	15	13.5	13	8	<b>78.5</b>
	4	24.5	13	13	13	8	<b>71.5</b>
<b>Cacao</b>	0	34.5	18	13.5	13.5	8.5	<b>88</b>
	1	33.5	18	13.5	13.5	8.5	<b>87</b>
	2	30.5	16.5	13	13	8.5	<b>81.5</b>
	3	26.5	13.5	13	13	8	<b>74</b>
	4	24	11.5	12.5	12	8	<b>68</b>
<b>LSD(P≤0.05 )</b>	<b>0.694</b>						

Table 5: Sensory evaluation for flavoured gels prepared by using ferrous sulphate during storage at 7±1 °C for up 28 days.

Flavour	Week	Flavour 40	Bitterness 20	Body 15	Texture 15	Appearance & colour 10	Total 100
FS - Melon	0	33	17.5	14	13.5	8.5	86.5
	1	32.5	17	14	13.5	8.5	85.5
	2	32	17	13.5	13.5	8	84
	3	30.5	16.5	13.5	13	7.5	80.5
	4	30	14.5	13	13	7	77.5
Mint	0	32	16.5	13.5	13.5	8.5	84.5
	1	32	16.5	13.5	13.5	8.5	84
	2	31.5	16.5	13.5	13.5	8	83
	3	29.5	15.5	13.5	13	8	79.5
	4	25.5	14	13	13	7.5	73
Cacao	0	35.5	17.5	13.5	13	8.5	88
	1	35	17.5	13.5	13	8.5	87.5
	2	33.5	17	13	12.5	8.5	84.5
	3	30.5	15.5	12	12	8	78
	4	24.5	13.5	11.5	12	7.5	69
LSD(P≤0.05) )	0.536						

## CONCLUSION

Ferrous milk protein gels were produced from skim milk using heat treatment and ferrous salts addition (18 mM). Sensory evaluation of flavoured gels was higher than the non-flavoured and textural properties of flavoured gels were better also.

## REFERENCES:

1. Amatayakul, T., F. Sherkat and N.P. Shah (2006) "Physical characteristics of set yoghurt made with altered casein to whey protein ratios and EPS producing starter cultures at 9 and 14% total solids." Food Hydrocolloids 20: 314-324.
2. Becker, T. and Z. Puhan (1989) "Effect of different process to increase the milk solids non-fat content on the rheological properties of yoghurt." Milchwissenschaft. 44: 626-629.
3. Bonczar, G., M. Wszolek and A. Siuta (2002) "The effects of certain factors on the properties of yoghurt made from ewes milk." Food Chemistry 79: 85-91.
4. Boye, J.I., Alli, I., Ramaswamy, H., Raghavan, V.G., 1997. Interactive effects of factors affecting gelation of whey proteins. J. Food Sci. 62, 57–65.
5. Conochie, J. & Sutherland, B.J. (1965). The nature and cause of seaminess in Cheddar cheese. Journal of Dairy Research, 32, 35–44.

6. De wit, J. N.; Hontelez-Backx, E. and Adamse, M. (1988). Evaluation of functional properties of whey protein concentrates and whey protein isolates.3. Functional properties in aqueous solution. *Neth. Milk Dairy Journal*, 42, 155-172.
7. Fox, P. F. and McSweeney, P.L.H. (1998). *Dairy Chemistry and Biochemistry*. Blackie Academic and Professional Publishers, London, New York. pp. 347-378.
8. Harte F., L Luedecke., B. Swanson and G.V. Barbosa-Canovas (2003). Low fat set yoghurt made from milk subjected to combinations of high hydrostatic pressure and thermal processing. *J. Dairy Sci.* 86:1074-1082.
9. Kebary, K. M. K. and Morris, H. A. (1990). Effect of homogenization of reconstituted nonfat dry milk and butter oil mixtures on curd formation and characteristics. *Cultured Dairy Products Journal*, 25:12-18.
10. Lee, Y.H., Marshall, R.T., 1984. Strength of rennet curd made from milk and chemically modified soy proteins. *J. Dairy Sci.* 67, 263–269.
11. Lucey, J.A., Tamehana, M., Singh, H., Munro, P.A., 1998a. Effect of interactions between denatured whey proteins and casein micelles on the formation and rheological properties of acid skim milk gels. *J. Dairy Res.* 65, 555–567.
12. Lucey, J.A., Tet Teo, C., Munro, P., Singh, H., 1998b. Microstructure, permeability and appearance of acid gels made from heated skim milk. *Food Hydrocolloids* 12,159–165.
13. Mangino, M. E. (1984). Physicochemical aspects of whey protein functionality. *Journal of Dairy Science*, 67, 2711-2722.
14. Puvanenthiran, A., Williams, R. P. W., and Augustin, M. A. (2002). Structure and visco-elastic properties of set yoghurt with altered casein to whey protein ratios. *International Dairy Journal*, 12(4): 383-391.
15. Ramasubramanian, L. (2013). The interaction of ionic calcium and milk proteins during heat treatment. PhD thesis, The University of Queensland, Brisbane, Australia.
16. Ramasubramanian, L.; D'Arcy, B. R. and Deeth, H. C. (2012). Heat-induced coagulation of whole milk by high levels of calcium chloride. *International Journal of Dairy Technology*, 65, 183-190.
17. Ramasubramanian, R., D'Arcy, B.R., Deeth, H.C., Oh, H.E., 2014. The rheological properties of calcium-induced milk gels. *J. Food Eng.* 130, 45–51.
18. Sievanen, K., Huppertz, T., Kelly, A.L., Fox, P.F., 2008. Influence of added calcium chloride on the heat stability of unconcentrated and concentrated bovine milk. *Int. J. Dairy Technol.* 61, 151–155.
19. Zirbel, F. and Kinsella, J. E. (1988). Factors affecting the rheological properties of gels made from whey protein isolate. *Milchwissenschaft*, 43 ,691–694.