STUDYING THE COMBINED EFFECTS OF THE PARTICLE SIZE & STORAGE CONDITIONS ON MILK POWDER AGING

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Abstract – *Whole milk powder is an important food substance worldwide with a huge number of consumers across the globe. The research is based on the aging of the milk powder on the functional properties viz. wettability, water activity, lactose crystallization & free fat content. The effect of the functional parameters on the milk powder with different particle size and relative humidity was observed over the time span of eight weeks. The selected particle size are 450 µm & 180 µm & the selected relative humidity is 65% & 80%. Ammonium nitrate & ammonium sulphate are used to maintain the relative humidity of 65% & 80% respectively in the desiccator. The results from this research stated that the milk powder with particle size of 450 µm & relative humidity of 65% was aged faster as compared with the other particle sizes of the milk powder.*

Key Words: whole milk powder, functional properties, particle size, relative humidity, wettability, water activity, lactose crystallization, free fat content

1. INTRODUCTION

Milk & milk powder are the two most essential dairy food products which are consumed by almost every living being whether humans or animals, as they are known to contain the high nutritional values of fat, soluble proteins, lactose present in the form of carbohydrate and other essential minerals and vitamins (Thomas, Scher, Desobry-Banon, & Desobry, 2004). In recent years, the production of milk powder has increased rapidly & thus, it is becoming important to maintain the functional properties & nutritional value of the milk powder as these values are entirely unpredictable & varies during production (Tehrany & Sonneveld, 2010).

Milk powder is a type of dairy product which is manufactured by evaporating the water content from the liquid milk using several drying processes until the fine powder is obtained. Raw milk has high moisture content due to which the shelf life of the raw milk is relatively shorter. To increase the shelf life of raw milk without compromising the quality & to reduce the volume of transportation and handling, the raw milk is converted into the milk powder (Kumar, 1990). Milk powder has a relatively higher shelf life in comparison with liquid milk and, the milk powder does not need refrigeration. Hence, the milk powder can be stored easily for months in atmospheric conditions (Rotronic Measurement Solutions, 2015). The first procedure to extend the shelf life of the milk was the fermentation, but as the production of the milk increases, drying came into existence (Thomas, Scher, Desobry-Banon, & Desobry, 2004). Freeze drying, bed drying, and shelf drying are some of the methods available which are useful in the manufacturing of the milk powder. Roller drying and spray drying are the two most common methods used in the production of the milk powder. However, the milk powder produced by using roller drying was not of good quality. The powder had an irregular shape with a wrinkled surface and rough edges which resulted in decreased packing efficiency and obstructions in free flow. Spray drying was introduced to produce the milk powder to overcome these problems and the process turned out to be the most common technique during the 1960s (Refstrup, 1995). By adopting the spray drying method, it was observed that the shelf life of the whole milk pwder and the skim milk powder increased in two years and three years respectively (Bylund, 1995; Carić, 1994).

The research will discuss the effects of various functional properties of milk powder like lactose crystallization, free fat content, wettability and water activity with aging. Not much work has been done on the topic before and hence, studying these effects with time is really important because milk powder is one of the most crucial substance consumed by everyone.

2. METHODOLOGY

Milk powder was sieved to obtain the two particle sizes i.e. 180 µm and 450 µm. The functional properties of the selected size of the milk powder were determined for the initial week. After that, the milk powder was kept in desiccators to maintain the relative humidity level of 65% and 80% using salts ammonium nitrate and ammonium sulphate respectively. The functional properties were measured for week 1, week 2, week 3, week 4, week 6 and week 8. All the experiments were performed in triplicates so that the precise observations can be obtained.

Following methods are used to determine the functional properties of the milk powder.

2.1 Free Fat Content

2 gm of sample was taken in a centrifuge tube and 15 ml of analytical grade pentane is added to the sample. The tube was shaken for 1 minute to suspend all insoluble particles. After that, the tubes were centrifuged using IEC clinical centrifuge. 2 ml of the clear supernatant liquid was pipette in Al weighing dishes and the dishes were placed in a water bath

maintained at a temperature of 80°C under fume hood. Since the pentane is highly flammable, it is safe to use the fume hood for drying of the liquid. Before keeping the liquid in Al weighing dishes, the initial weight of the dishes was recorded and after the liquid has been dried the final weight of Al dish was taken. The difference between the two weights is the measurement of the free fat content in the milk powder.Scanning Electron Microscopy (SEM) was used to compare the total fat content and the free fat content of the milk powder. Philips XL30S Field Emission Gun (FEG) microscope was used for the analysis of the fat content in the milk powder. The sample was mounted in the microscope and then a beam of back scattered electrons was allowed to hit the sample to obtain the microscopic image. The working distance needs to set by the user in order to get the focused image with all details. Therefore, the working distance is the most important factor in the SEM.

2.2 Lactose Crystallization

Lactose crystallization was measured using X-Ray powder Diffraction (XRD) by placing 1 gm of sample in the sample holder. The sample is exposed to the beam of X-rays produced by the XRD equipment and the spectrum was obtained on the computer with the help of imaging software. The amount of lactose crystallinity will be represented by the highest intensity X-ray and if the characteristic peak is not present, it means that the lactose is present in the amorphous form.

2.3 Wettability

400 ml of water maintained at room temperature was taken in the wettability beaker. 13 gm of the sample was transferred into a glass plate placed over the beaker. The glass plate was removed from the beaker. When the last fragment of the milk powder passes through the water, the time was recorded and the recorded time is the wettability time or wettability.

2.4 Water Activity

The water activity meter was calibrated to obtain the precise results. 1 gm of sample was placed in the sample holder which was further placed in the water activity meter. The meter analyses the sample and the value of water activity is displayed on the screen.

All the calculations of the data was carried out in the Microsoft Excel 365.

3. RESULTS & DISCUSSIONS

3.1 Wettability:

Table -1: Values of wettability of milk powder in triplicates for different weeks

Particle size: 180 μm and 450 μm
Relative humidity: 65% and 80%

Weeks	Wettability (in Seconds)			
	180 μm 80%	180 μm 65%	450 μm 80%	450 μm 65%
Week 0	3142.62	3142.62	996.66	996.66
	3164.61	3162.32	1013.02	1019.02
	3167.68	3172.14	1028.86	1034.86
Week 1	3091.96	3436.59	1521.87	1062.96
	3123.58	3473.64	1583.71	1149.68
	3128.76	3504.31	1604.57	1148.97
Week 2	3408.08	3860.72	1774.67	1559.02
	3423.32	3912.54	1782.3	1631.95
	3451.33	3952.67	1796.12	1646.01
Week 3	3785.83	4399.92	2228.61	2109.96
	3815.62	4454.63	2286.1	2179.56
	3843.62	4496.29	2339.32	2231.87
Week 4	4031.85	5005.43	2853.69	2735.58
	4065.64	5063.12	2932.87	2811.84
	4094.41	5109.09	2973.92	2867.23
Week 6	4403.24	5586.43	3473.38	3506.61
	4444.73	5517.12	3583.17	3577.74
	4476.76	5653.32	3602.21	3653.25
Week 8	5156.21	5972.17	4153.76	3901.73
	5173.97	6023.95	4282.31	4067.23
	5225.32	6111.31	4104.18	4063.12

Table 1 shows the values of wettability over week 0, week 1, week 2, week 3, week 4, week 6 & week 8. Experiments were conducted in triplicates in order to obtain the precise observations for a particular particle size and at a particular relative humidity. From the data, the standard deviation was calculated and is represented in table 2.

Table -2: Standard Deviation of the data in table 1

Standard Deviations				
Weeks	180 μm 80%	180 μm 65%	450 μm 80%	450 μm 65%
Week 0	11.16038928	12.27442146	13.14616632	15.67062078
Week 1	16.26481137	27.6874416	35.11665512	40.71388325
Week	17.91144	37.63941	37.63941	38.12804

2	947	758	758	247
Week 3	23.59644088	39.46294549	45.20837263	49.93611941
Week 4	25.56740677	42.40908262	49.89970808	53.9705292
Week 6	30.09712316	55.60634276	56.77786599	59.87443027
Week 8	29.30383403	57.41939045	75.06558303	77.06697967

The graph of the standard deviation on the ordinate and weeks on the abscissa was plotted to understand the effect of wettability on different conditions of the milk powder. The graph is represented as chart 1.

Chart -1: Standard Deviation values of wettability vs Weeks

It can be clearly seen from the chart that 450 µm (coarse) particle size at relative humidity 80% has the more wettability which means that at this condition the milk powder has more tendency to absorb water at the surface and after absorbing the maximum water the milk powder passes through the surface of the stable water which simply explains the extent of wettability (Sharma, Jana, & Chavan, 2012). Wettability is also dependent on the contact angle with the liquid and the solid particle. The smaller the angle, more the wettability & vice-versa (Schuck, Dolivet, & Jeantet, 2012). Due to larger surface area of the 450 µm particle size, the contact angle is smaller and hence, the larger wettability. Smaller value of relative humidity means the particle has more tendency to absorb water and hence, the milk powder with 450 µm particle size and a relative humidity of 80% has the highest wettability.

Also, the blue line from the chart indicates the lowest values of wettability which means that the smaller surface area of the 180 µm particle size will have larger contact angle with the surface of water and hence has the lowest wettability. Now, the relative humidity 80% means the powder has less tendency to absorb water from the surface and it will pass through the surface of water very easily with a shorter wait time. Therefore, the milk powder with particle size 180 µm and relative humidity 80% has the lower values of wettability.

3.2 Water Activity:

The amount of free water present in the food substance is the water activity which is accountable for the food safety and is responsible for all the chemical and biochemical reactions within the food particles. If the water activity is kept below 0.85, then most of the enzymes are inactivated (Safefood 360° , 2014).

Table -3: Values of water activity of milk powder in triplicates for different weeks

Particle size: 180 µm and 450 µm
Relative humidity: 65% and 80%

Weeks	**Water Activity (in %)**			
	180 μm 80%	180 μm 65%	450 μm 80%	450 μm 65%
Week 0	38.9	37.9	47.1	48.8
	38.2	37.9	47.6	48.2
	38.3	37.3	47.8	48.9
Week 1	44.9	42.4	52.5	53.3
	44.3	43.1	52	52.2
	45.3	42.9	51.7	53.3
Week 2	48	46.6	58.1	57.9
	48.5	45.9	58.6	57.2
	47.3	45.5	57.6	56.5
Week 3	50.7	50.7	62.8	62.2
	51.6	51.5	61.3	62.7
	51.9	50.1	62.4	61.2
Week 4	55	54.7	62.9	64.7
	55.6	53.4	61.3	64.1
	56.5	54.4	61.9	65.8
Week 6	62.2	62.8	68.3	68.4
	62.9	62.1	67.1	68.7
	61.3	61.2	68.6	67.1
Week 8	68.1	68.8	70.3	73.7
	67.3	70.3	71.9	72.2
	68.9	69.8	71.5	73.9

Table 3 shows the values of water activity obtained experimentally using the water activity meter over week 0, week 1, week 2, week 3, week 4, week 6 & week 8. Experiments were conducted in triplicates in order to obtain the precise observations for a particular particle size and at a particular relative humidity. From the data in table 3, the standard deviation was calculated and is represented in table 4. Microsoft Excel 365 was used to calculate the standard deviation of the data.

Table -4: Standard Deviation of the data in table 3

Standard Deviations				
Weeks	180 μm 80%	180 μm 65%	450 μm 80%	450 μm 65%
Week 0	0.309120617	0.282842712	0.294392029	0.309120617
Week 1	0.410960934	0.294392029	0.329983165	0.518544973
Week 2	0.492160769	0.454606057	0.40824829	0.571547607
Week 3	0.509901951	0.573488351	0.63420992	0.623609564
Week 4	0.6164414	0.555777733	0.659966329	0.703957069
Week 6	0.65489609	0.65489609	0.64807407	0.694422222
Week 8	0.653197265	0.623609564	0.679869268	0.758653778

The graph of the standard deviation on the ordinate or the y-axis and weeks on the abscissa or the x-axis was plotted using Microsoft excel 365 to understand the effect of water activity on different conditions of the milk powder. The graph is represented as chart 2.

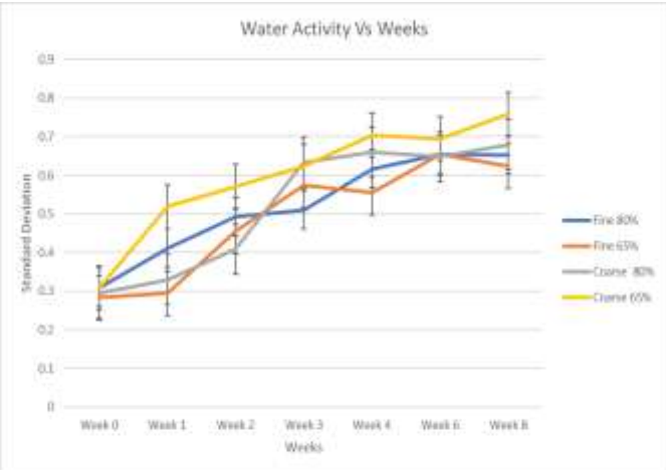

Chart -2: Standard Deviation values of water activity vs Weeks

From chart 2, it can be seen that the milk powder with 180 μm particle size and relative humidity 65% had the lowest water activity at week 0 and then the value of the water activity goes on increasing as the time increases but there was a sudden increase in the water activity value was observed from week 1 to week 3 and after that very small change was noticed which means that after a certain time the water activity value does not vary exponentially. For the milk powder with particle size 450 μm and relative humidity 80%, it can be observed that the week 3 has the highest water activity value and then it goes on decreasing as the time increases and an exponential increase was noticed over week 2 and week 3 but from week 3 to week 8, the water activity was almost constant as there was very small change in the values of the water activity which simply means that as the time increases, the value of water activity tends to be constant. For the particle size 180 μm and relative humidity 80%, an exponential increase was noticed from week 0 to week 2 and almost a constant value of water activity was observed from week 2 to week 3 and again an exponential increase was observed from week 3 to week 6 but the water activity value showed a negligible change from week 6 to week 8. For the milk powder with particle size 450 μm and relative humidity 65%, it can be observed that the value of water activity gradually increased from week 0 to week 1 and from week 1 to week 4, an exponential increase can be noticed and the value does not seem to be constant at week 8. At week 8, there was an increase in the value. This milk powder showed the highest value of water activity at all the weeks except the week 3.

Therefore, the milk powder with particle size 450 μm and relative humidity 65% has the highest values of water activity and the lowest values of water activity was shown by the milk powder with particle size 180 μm and relative humidity 65%.

3.3 Lactose Crystallization:

The rate of lactose crystallization is generally enhanced by the increasing temperature and increased relative humidity (Thomas, Scher, Desobry-Banon, & Desobry, 2004). The lactose crystallization of milk powder occurs when the milk powder turns in glassy state and the crystal changes from amorphous form to crystalline form (Thomsen, Lauridsen, Skibsted, & Risbo, 2005).

The charts for the lactose crystallization was obtained using XRD technique. Figure 1 shows the trend of lactose crystallization of milk powder having particle size 180 μm and relative humidity 65% whereas the figure 2 shows the trend of the lactose crystallization of milk powder with the particle size of 180 μm and the relative humidity of 80%.

Figure 3 represents the crystallinity of lactose of milk powder with the particle size of 450 μm and the relative humidity of 65% and the figure 4 represents the lactose crystallization of milk powder with the particle size of 450 μm and the relative humidity of 80%.

From figure 1, it can be stated that the milk powder does not change from amorphous to crystalline stage easily because of the deviation of the points from the possible trend of the lactose crystallinity. Week 2 and week 8 were the outliers which means that there is no lactose crystallinity in those two weeks. The lactose crystallization of milk powder with particle size 180 μm and relative humidity 65% occurs mainly in week 1, week 4 and week 6. At week 1, week 4 and week 6, the points fall in the confidence interval of 95%. As the outlier for week 2 is above the possible trend, so it simply means that the rate of lactose crystallization is very fast in the milk powder.

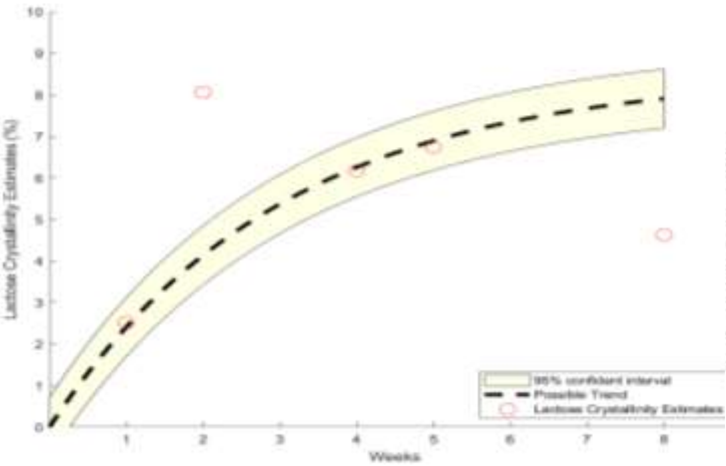

Fig -1: Trend of lactose crystallization with particle size 180 μm and relative humidity 65%

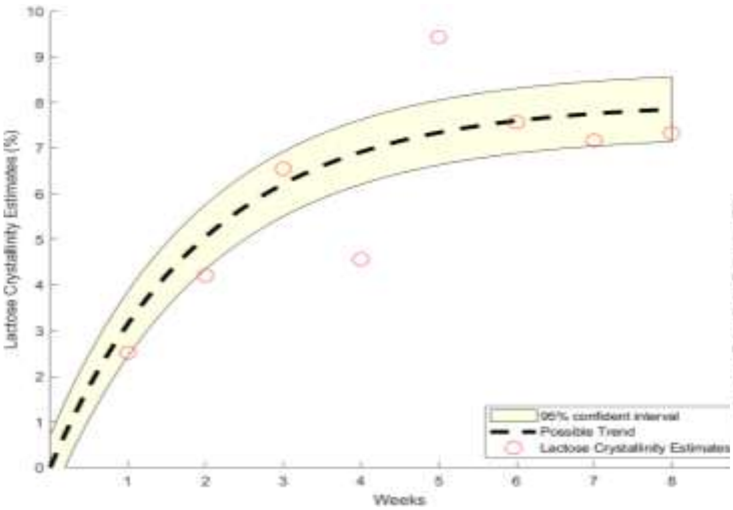

Fig -2: Trend of lactose crystallization with particle size 180 μm and relative humidity 80%

Figure 2 shows a good trend of lactose crystallization and it can be said that there are only 2 outliers which signifies that these outliers do not contribute towards the state change of milk powder from the amorphous state to crystalline state. The maximum lactose crystallization occurred at week 3 and week 6 which is followed by week 1, week 2 and week 8. The lactose crystallinity points at week 1, week 2 and week 8 lies in the confidence interval of 95% which means that the desired crystallinity of lactose can be achieved easily and the process is economical. Since the outlier at week 4 is below the possible trend, so it signifies that the milk powder tries to remain in amorphous state and it is very difficult for the milk powder to convert into crystalline form. Week 6 outlier is well above the possible trend, it can be due to the fact that either the powder is already in crystalline form or can be easily converted to crystalline form to achieve the lactose crystallinity.

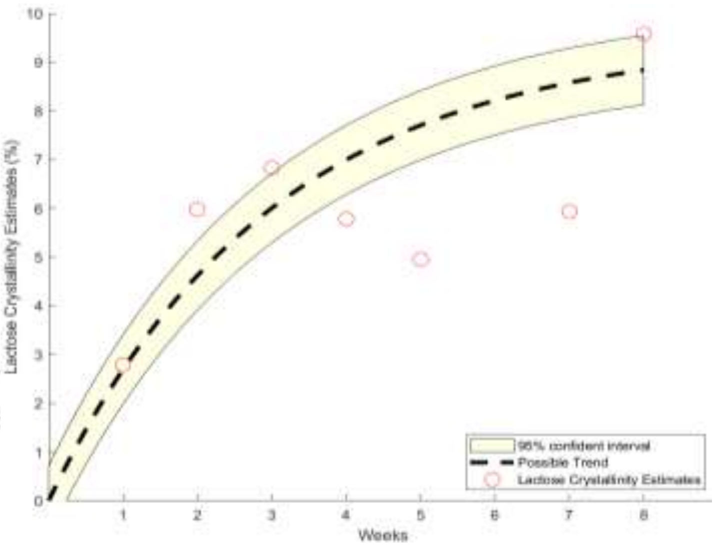

Fig -3: Trend of lactose crystallization with particle size 450 μm and relative humidity 65%

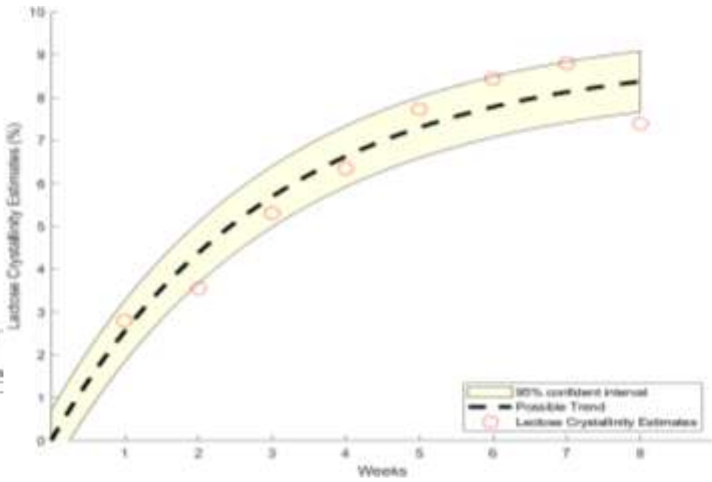

Fig -4: Trend of lactose crystallization with particle size 450 μm and relative humidity 80%

Figure 3 signifies that there is very less lactose crystallization of milk powder with particle size 450 μm and relative humidity 65%. It is because of the maximum outliers present in the XRD graph. The maximum crystallinity of lactose was observed only in week 1 and very less crystalline form of milk powder was observed in week 3 and week 8. AT week 1, week 3 and week 8, the points falls in the region of the confidence interval of 95% which signifies that the lactose crystallization can be obtained easily. At week 4, week 5, and week 6, three outliers were obtained which were well below the possible trend line showing that it is very difficult for the milk powder with particle size of 450 μm and relative humidity 65% to convert to crystalline form from the amorphous form. But, at week 4, the milk powder is near to the possible trend line and tries to achieve the confidence interval of 95% and it can be said that it is possible at week 4 that some crystallinity of lactose may be observed. At week 2, one more outlier is obtained which is well above the possible trend of the lactose crystallization which simply signifies that the milk powder is available in the crystalline form only.

Figure 4 shows the best possible trend of the lactose crystallization for milk powder with particle size 450 μm and the relative humidity of 80% because most of the points lies on the possible trend line and it has only one outlier which is also very close the possible trend line and almost all the point lies inside the confidence interval of 95%. Week 1, week 3, week 4, and week 6 has the maximum lactose crystallization whereas at week 2, there is very less crystallinity of lactose because point is little bit deviated from the possible trend line but lies in the confidence interval of 95% in the XRD graph. Also, at week 8, an outlier is obtained which is very close to the possible trend line and it tries to achieve the confidence interval of 95% which simply tells that the milk powder can easily be converted to the crystalline form from the amorphous state.

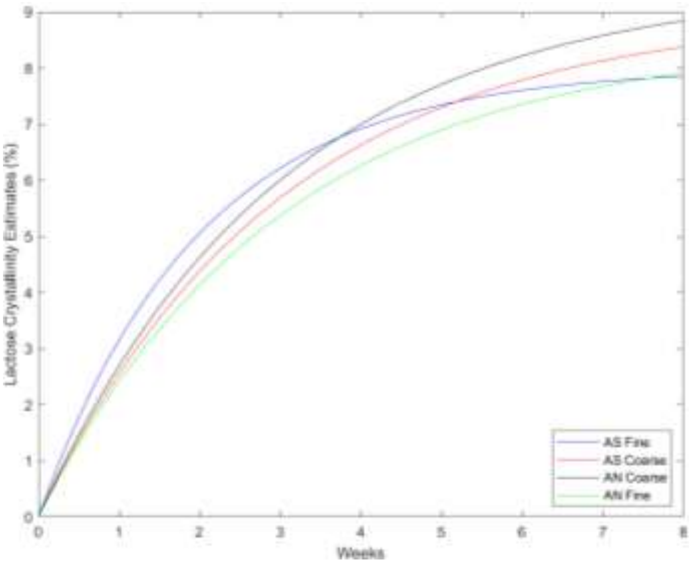

Fig -5: General trend of lactose crystallization for different conditions of the milk powder

From figure 5, it is clearly seen that the value of lactose crystallization increased gradually over the weeks for all the milk powders at different conditions. The milk powder with the particle size of 180 μm and the relative humidity of 65% (AN fine) had the lowest lactose crystallinity and it is due to the fact that lactose crystallization increases with the increase in temperature and the relative humidity. For the milk powder with the particle size 180 μm and the relative humidity of 80% (AS fine) started from 0% at week 0, but increased gradually as the time increases and showed the highest value till week 4 but after week 4 the crystallinity almost became constant and achieved the same value as of the milk powder with particle size 180 μm and a relative humidity of 60%. For the milk powder with the particle size 450 μm and the relative humidity 80% (AS coarse), the crystallinity of lactose increased gradually and was second highest after week 6. For the milk powder with particle size 450 μm and the relative humidity of 65% (AN coarse), it can be stated that it showed the highest value of lactose crystallization after week 4. Also, at week 4, AS fine and AS coarse milk powders, both show the same rate of lactose crystallization.

3.4 Free Fat Content:

Table -5: Values of free fat content of milk powder in triplicates for different weeks

Particle size: 180 μm and 450 μm
Relative humidity: 65% and 80%

Weeks	Free Fat Content			
	180 μm 80%	180 μm 65%	450 μm 80%	450 μm 65%
Week 0	0.0491	0.0421	0.045	0.044
	0.0433	0.0433	0.042	0.041
	0.0481	0.0481	0.038	0.0367
Week 1	0.0524	0.0488	0.0492	0.0395
	0.0481	0.0409	0.0552	0.0402
	0.0554	0.0411	0.0556	0.0322
Week 2	0.0598	0.0478	0.0586	0.0356
	0.0551	0.0408	0.0527	0.0326
	0.0623	0.0402	0.0521	0.0266
Week 3	0.0639	0.0601	0.0604	0.0497
	0.0612	0.0539	0.0673	0.0593
	0.0687	0.0518	0.0656	0.0563
Week 4	0.0696	0.0586	0.0675	0.0585
	0.0629	0.0638	0.0601	0.0625
	0.0698	0.0699	0.0587	0.0694
Week 6	0.0674	0.0711	0.0718	0.0714
	0.0717	0.0792	0.0795	0.0668
	0.0765	0.0676	0.0689	0.0795
Week 8	0.0742	0.0702	0.0751	0.0723
	0.0692	0.0797	0.0702	0.0648
	0.0785	0.0691	0.0623	0.0784

Table 5 shows the values of water activity obtained experimentally using the water activity meter over week 0, week 1, week 2, week 3, week 4, week 6 & week 8. Experiments were conducted in triplicates in order to obtain the precise observations for a particular particle size and at a particular relative humidity. From the data in table 5, the standard deviation was calculated and is represented in table 6. Microsoft Excel 365 was used to calculate the standard deviation of the data.

Table -6: Standard Deviation of the data in table 5

Standard Deviations				
Weeks	180 μm 80%	180 μm 65%	450 μm 80%	450 μm 65%
Week 0	0.002531578	0.002592296	0.002867442	0.002995923
Week 1	0.002995923	0.003677862	0.002927266	0.00361755
Week 2	0.002984776	0.00344996	0.002932955	0.003741657
Week 3	0.003101612	0.003523571	0.002935227	0.004009988
Week 4	0.00320659	0.00461808	0.003860915	0.004502098
Week 6	0.003716928	0.004858212	0.004472881	0.005249974
Week 8	0.003800292	0.004758851	0.005273203	0.005561974

Chart -3: Standard Deviation values of free fat content vs Weeks

The graph of the standard deviation on the ordinate or the y-axis and weeks on the abscissa or the x-axis was plotted using Microsoft excel 365 to understand the effect of water activity on different conditions of the milk powder. The graph is represented as chart 3.

From chart 3, it can be clearly observed that the milk powder with particle size 450 μm and relative humidity of 65% has the highest values of free fat content over the time but the milk powder with particle size 180 μm and relative humidity of 65%, there was an exponential increase over week 1 and then remained constant from week 1 to week 3 and gradually increased over week 4 to week 8. The milk powder with particle size 450 μm and relative humidity of 80%, the free fat content was constant till week 3 and then increased exponentially till week 8. For the milk powder with particle size 180 μm and relative humidity of 80%, the free fat content was constant till week 4, but there was a gradual increase from week 4 to week 6 and then became almost constant over week 6 to week 8.

Scanning Electron Microscopy (SEM) Images will be used to compare the free fat content in coarse (450 μm) and .

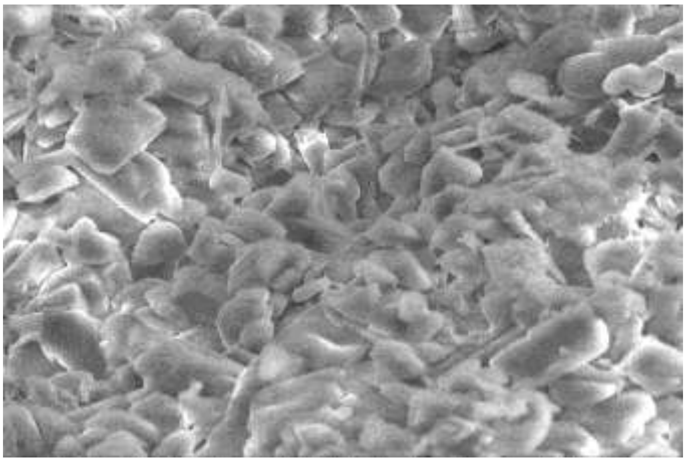

Fig -6: SEM image of free fat for coarse milk powder (450 μm)

The free fat content SEM image for coarse (450 μm) and fine powder (180 μm) is represented as figure 6 & figure 7 respectively. There is no free fat in the milk powder which signifies that the fat content in the milk powder is still not broken into small pieces to form the free fat.

From the figure 6 & figure 7, it can be clearly seen that the lumps of fat are present which is simply the total fat of the milk powder. Over the time, this fat will break, which will be termed as the free fat content. The only difference in both the images is that for the coarse particle size the lumps are bigger in size and for the fine particle size, smaller sized lumps are formed for the fat content.

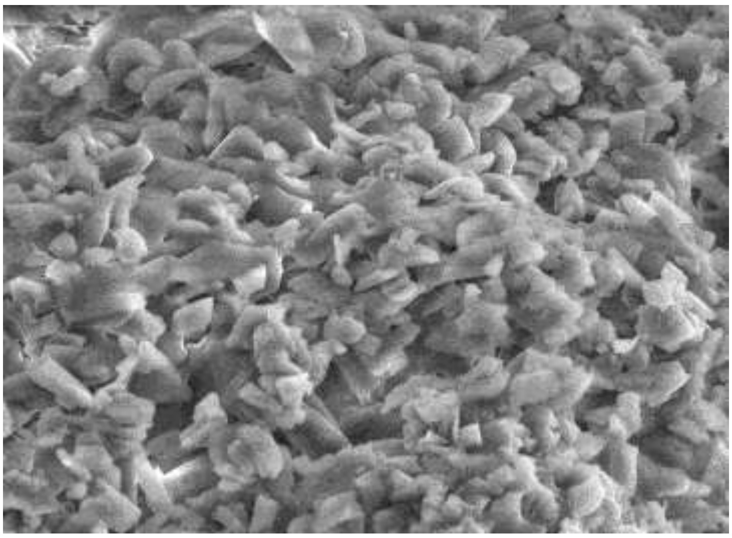

Fig -7: SEM image of free fat for coarse milk powder (180 μm)

4. CONCLUSIONS

It can be concluded that wettability, water activity and the free fat content was maximum for the milk powder with particle size 450 μm and the relative humidity of 65%. The lactose crystallization was maximum for the milk powder with the particle size 450 μm and the relative humidity of 65% but till week 4 the milk powder with particle size 180 μm and relative humidity of 80% had the maximum crystallinity of lactose and from week 4 to week 8 the value was almost constant.

Therefore, the milk powder with the particle size of 450 μm and relative humidity of 80% was aged maximum over 8 weeks. Due to time constraints, the experiments were not performed for the further weeks. The future research could be done by performing the experiments over a larger period of time.

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BIOGRAPHIES

Being impromptu is what relates me but giving my work in an organized manner is my attitude. Working on new things and discovering them is my passion. Perseverance and being practical is what I work upon to define me.

Patience, attitude and discipline are the traits that define me. Hardwork and enthusiasm to work is my nature. Exploring and working on new things and gifting them to the world in the form of writing is my passion.