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Effect of Sunlight Exposure on Spectroscopic, Thermal, Crystalline, Morphological Properties and Thermal Degradation Profile of Potato Starch

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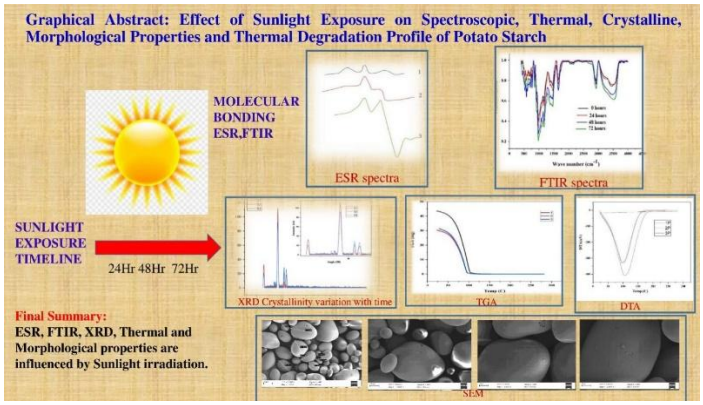
ABSTRACT
Exposure of potato starch to sun light causes changes in chemical structure accompanied with changes in physical properties (crystalline, morphology and thermal) as well as health concern. In the present studies an attempt has been made to nvestigate the changes by spectroscopic (FTIR & ESR), crystalline (XRD), thermal (TGA& DTA), morphology (SEM) techniques. The spectroscopic results confirm cleavages of chemical groups / bonds and molecular chains forming free radicals. XRD data indicate decrease of degree of crystallinity though diffraction pattern remain the same. Williamson – Hall method is used to estimate crystalline width and lattice distortion parameters. Thermal degradation of sunlight exposed PS followed two stage degradation process though profiles are different. Decrease in weight loss is almost same up to 40^0C; the decrease of weight loss increased up to 60 – 80^0C and finally the weight loss decrease is rapid in between 80 -100^0 C. Activation energy corresponding thermal degradation is evaluated. DTA profile of starch appears to have broad endothermic peak centred around 100^0C together with hidden component around 50 -70^0 C. The latent peak correspond to gelatinization of starch: while the broad peak is attributed to dissociation of amylase component. Morphology of starch granules is influenced by sunlight exposure. The less exposed granular surface appears to be smooth when compared to rough and broken surface of high exposed granules. The crack size and pore size increase with time of exposure reflecting sunlight induced damage of starch granule. Sunlight exposure of potato starch has an adverse effect on human health. Formation of green colour on the skin of PS is an indication for this. Formation of toxic materials like glycoalkoloids and nitriles is an indication for green color development of PS. In the present studies the author made an attempt to identify / quantify these components.

Graphical Abstract:



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INTRODUCTION:

Potatoes are an important source of food material due to presence of carbohydrates, dietary fibre, proteins and minerals. Therefore, potato starch is one of the important class among the starches having B – type crystalline structure and occupy next place to rice and wheat starches with respect to cultivation and consumption. During its use or processing the PS is invariably exposed to sunlight and therefore these effects are to be considered for any application. In this context the authors summarize some important reports on PS in recent times.

S.No	Title	Result	References
1	Effect of UV B irradiation on Corn &potato starches	UV B irradiation causes minor changes. Functional properties are mildly effected. ESR studies suggest depolymerization	1
2	UV exposure on starch films	Dehydration occurs Optical,mechanical Physiological properties are measured	2
3	Effect of UV-C irradiation on Physico-chemical characterization of Potato tubers	UV LED lamp irradiation for 30 minutes. Weight loss observed and anti-oxidant activity decreases	3
4	EPR study irradiated starch – Dose assessment	Dose assessment be done with starch as medium	4
5	Effect of light – PS based bio-plastics	Edision type Incandiscent lamp used for irradiation	5
6	The structural properties of different starches exposed to UV irradiation	Good photo stability is observed for PS & WS. Water loss was observed and main chain scission was observed	6
7	Response/ effect of Starch granule to microwavesradiation	Granular size changed Diameter and surface area are effected.	7
8	Free radical formation in UV and gamma irradiated Cassava starch	Free radical formation is compared. ESR studies suggest free radicals is same in both starches and irradiations.	8
9	Effect UV C irradiation on mechanical & physiological properties of potato tuber.	mechanical Physiological and textural properties are reported to changed on irradiation	9
10	Effect of low doseof UV B irradiation on purple potato starches	UV B irradiation on purple potato starch, effect of Physiological &Biological Changes are reported.	10

The starches suffer from the process of degradation during their applications in different areas and or during the time of processing like heating, mechanical (grinding / crushing), photo degradation, or high energy treatment (gamma, EB or X-rays). All these treatments initiate change of chemical structure followed by changes in physical, thermal and crystalline properties. The changes include i) stability enhancement [11]. ii) nutritional risk [12] iii) generation of stable ESR signal resulting dosimetric applications.

SCOPE OF THE PRESENT STUDIES:

1 Sunlight exposure plays an important role in altering Physico- chemical properties of PS. Therefore, an attempt has been made to investigate chemical structure change, free radicals produced, changes in crystalline properties, surface morphology and variations thermal behaviour.
2 Sunlight exposure has some ill effects over its food consumption due to the formation glycoalkoloids and nitriles. An attempt has been made to probe this in the present studies.
3 Sunlight exposure is expected to bring changes in crystalline properties like degree of crystallinity, crystallite size & distribution and lattice distortion. These aspects have been discussed in the present studies.
4 Effect of sunlight exposure on thermal degradation profiles of PS has been investigated under different conditions.

MATERIALS & METHODS:

Potato starch in the form of white powder is purchased from Sigma Aldrich used as it is without any further medication. Sunlight exposure is undertaken on average weathering day. ESR spectra are recorded on BRUKER Model spectrometer operating at X –band frequencies and 100 K Hz modulation. FTIR spectra are recorded on PERKIN – Elmer spectrometer for pellets of PS mixed with K Br. XRD spectra are recorded on SHIMADZU XRD – 7000 diffractometers. TGA / DTA thermo grams are recorded on SHIMADZU DTG -60S with a heating rate of 10^0 C / minute. SEM micrograms are recorded on HITACHI S – 3700 N microscope for coated samples of starch under different conditions.

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RESULTS & DISCUSSION:

ESR STUDIES PHOTO DEGRADATION:

ESR spectra of PS exposed to sunlight for 24, 48 and 72 hrs are as shown in Figure 1. As expected intensity of the low exposed sample is less than the high irradiated PS. Formation of free radicals in starches is generally considered to be associated with cleavage of either 1 – 6 or 1 – 4 glycosidic bonds [13,14].

Spectral characteristics of sunlight exposed PS are as listed in table 1

The spectral features indicate number of hf lines, overall intensity (free radical concentration) gradually increase with radiation dose. As radiation dose / exposure time more energy is deposited causing more chain cleavages. The spectral shape suggest presence of more than one free radical as reported previously [15]. The gradual decrease of g-values indicate the shift of free radical centre from unstable state to stable state [16,17] formation and stabilization of persistent free radicals the rising methane changed the free radical type or free radical change by temperature. The low intensity and ill resolved line shape is thought to associated with crystallinity and inter-molecular hydrogen bonding of starch [18]. The hydrogen bonding acts as a mantle / crystalline region so that the low radiation (line sunlight) are insufficient cause chain cleavages resulting in ill-resolved spectra. The results are in consistent with the reports of spectral resolution dependency on radiation and dose rate effects [15] The results are supported from FTIR and XRD data.

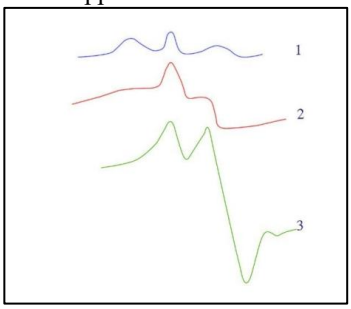

Figure1: ESR spectra of sunlight exposed PS [1-24 hours,2-48 hours,3- 72 hours.]

Table 1: Spectral characteristics of sunlight exposed PS

S No	Time of irradiation	Hyperfine lines	spread	g – value	Intensity A U
1	24 hrs	1-3	50 G	2.2951768000	10
2	48 hrs	3	60 G	2.1081199400	20
3	72 hrs	3-5	80 G	2.1069723473	50

FTIR STUDIES:

FTIR spectra of Sunlight exposed PS to different times are as shown in Figure 2. The spectra / absorption bands represent chemical structure / groups present in starch. The band positions and the assignments are as listed in Table2. More prominent bands are 3400 – 3300 cm^{-1} corresponding to --, 3250 – 3100 cm^{-1}, corresponding to O – H / H – H [hydrogen bonding], 2900 – 2800 cm^{-1} corresponding toC – H of CH_2 / CH_3 groups [18, 19].

The following conclusions can be drawn from FTIR data

1. Intensity of 3450 cm^{-1}absorption band decreased with exposure time suggesting decrease of symmetric OH groups or intermolecular hydrogen bonds. [20,21] and suggest removal of water from starch on irradiation.
2. Intensity of 3250 cm^{-1} absorption band also decreased with exposure linked with cleavage of O – H asymmetric bonds
3. The presence of 1650 cm^{-1} absorption band may be due to bound water and or Glykoalkoloids whose formation is discussed later
4. The 2200 – 2300 cm^{-1} absorption band is not characteristic of starches but it indicates the coexistence of material like nitrile.

Table 2: FTIR absorption bands of sunlight exposed PS under different conditions.

S.No	Time / Band position cm^{-1}			Assignment
	24 hours	48hours	72 hours	
1	3449	3449	3442	O – H symmetric & H – H bonding
2	3250	3250	3250	O-H Asymmetric
3	2926	2926	2926	CH_2/CH_3 groups
4	2870	2870	2870	CH_2
5	1650	1650	1650	Glcoalkoloids / water
6	1465	1465	1466	CH_2 / CH_3
7	1350	1350	1350	CH_2/ CH
8	1240	1240	1240	C – H , O - H
9	978	978	977	C – O
10	859	859	859	CH_2 / CH_3
11	765	765	765	Pyranoid ring

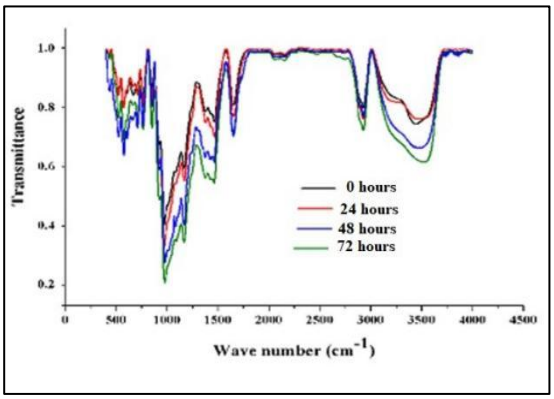

Figure 2: FTIR spectra of sunlight exposed PS.

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XRD STUDIES OF PHOTO DEGRADED PS

X-ray diffractograms of PS sunlight exposed to different times are shown as curves Figure 3a (crystalline peaks) and 3b crystalline peaks superposed on amorphous hallow).

XRD of non-irradiated PS shown peaks centred around 2θ = 5.5, 15.15, 17.7,22.1, 24.05 and 26.35 degrees. Due to sunlight exposure the peak intensities and positions shifted / decreased reflecting the shifting of crystal planes followed by lattice distortion. To investigate it further, the inter-planar distance between different crystalline planes of PS have been calculated using Bragg equation assuming λ for Kα is 1.54 nm and first order diffraction. Williamson Hall method is employed to calculate crystal damage and lattice distortion parameter [21].

Table 3: XRD data of non-irradiated and sunlight exposed PS.

Peak number	Peak Positions			
	Non-irradiated	Irradiated		
		24hours	48 hours	72 hours
P1	15.15 (3.793051)	15.10(3.805464)	14.95(3.876419)	14.9 (3.888780)
P2	17.7 (5.006884)	17.7 (5.006884)	17.7(5.006884)	17.7(5.006884)
P3	19.8(2.952203)	19.7(2.966566)	19.6(2.981070)	19.5(2.988465)
P4	22.1(2.658019)	21.87(2.684563)	21.70(2.704603)	21.6(2.716504)
P5	24.05(2.941092)	24.0(2.458633)	23.9(2.468282)	23.8(2.478069)

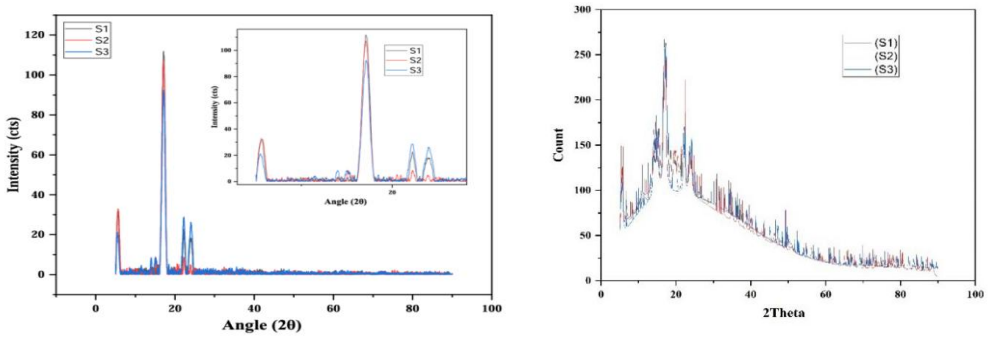

Figure 3: XRD of sunlight exposed PS at different times.

Measurment of Degree of Crystallinity (DOC):

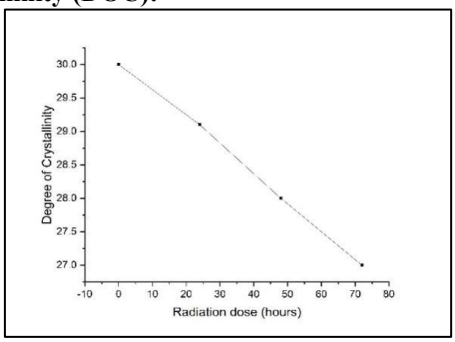

Figure 5: Variation of DOC against exposure time.

Sunlight exposure is expected vary DOC though overall shape of diffractogram does not change. The DOC values are calculated as described [22]. Variation of DOC against exposure time is as shown in Figure 5.

ESTIMATION OF CRYSTALLINE SIZE DISTRIBUTION & LATTICE DISTORTION:

Exposure of PS to sunlight is expected to cause changes in crystallize size distribution and lattice distortion as reflected from the broadening and shifting of XRD peaks. Williamson – Hall method is used to estimate both the quantities as described earlier [23,24]. Briefly the values of Sinθ and βCosθ are calculated and plotted as shown in Figure 6. From the graphs the values of crystallite size distribution and lattice distortion are evaluated at different exposure times and their variations are as shown in Figure 7. The sunlight exposure causes changes in crystalline / double helical structure of starch molecules and are thought to associated with the observed crystalline damage (25,26).

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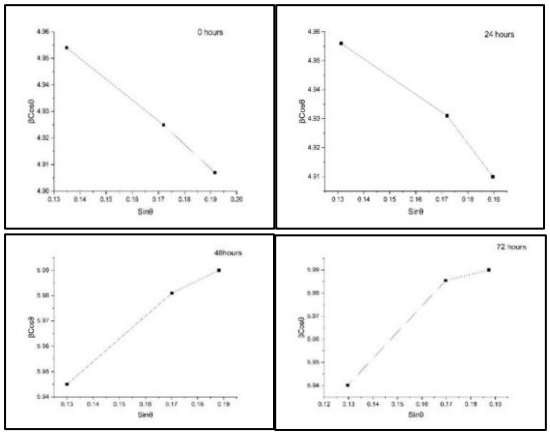

Figure 6: Variation of sinθ Vs βcosθ for different exposure times.

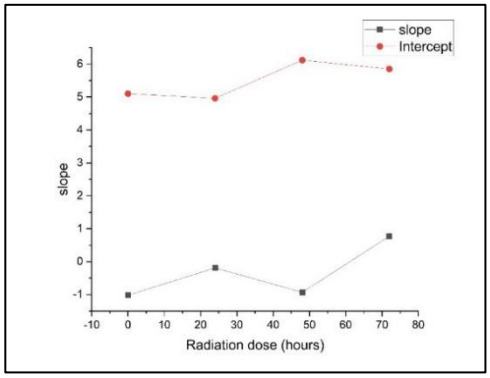

Figure 7: Variation of crystallize size and lattice distortion with exposure time.

THERMAL DEGRADATION BEHAVIOR OF SUNLIGHT EXPOSED PS

Thermo gravimetric curves of PS exposed to sunlight for different times are shown in Figure 8. The curves indicate two stage thermal degradation of PS. The first stage corresponding to release of bound / absorbed water (Region I), which normally occur around 20 – 100^0 C: while the second stage occurs above 100 – 150^0 C and corresponds to main decomposition of starch caused by cleavage of glycosidic bonds and degradation of amylose and amylopectin chains [27,28]. Due to sunlight exposure both the decomposition temperature ranges were shifted to low temperatures. Activation energy corresponding thermal degradation is evaluated by differential method [29]. The method require evaluation of values of $\frac{\Delta \ln(w)}{\Delta\left(\frac{1}{T}\right)}$ and $\frac{\Delta \ln dw/dt}{\Delta\left(\frac{1}{T}\right)}$

A plot of Y against X is drawn as shown in Fig 9, which intersects Y –axis: from which the value of activation energy is evaluated as 60 KJ / mol.

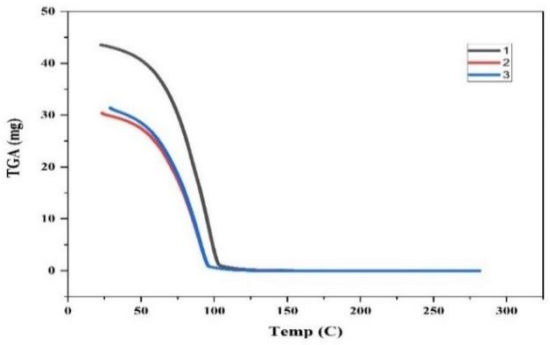

Figure 8: Thermogravimetric profiles of sunlight exposed PS

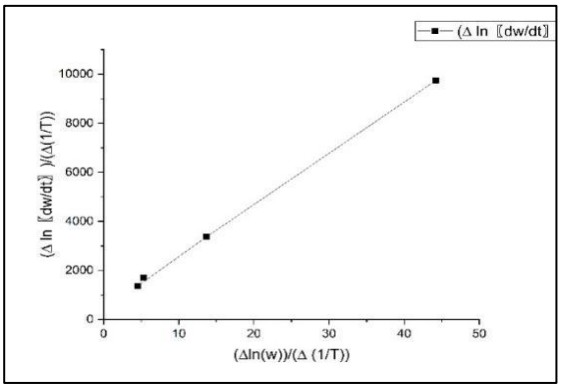

Figure 9: Evaluation of activation energy by differential method.

DIFFERENTIAL THERMAL ANALYSIS

Differential thermal analysis (DTA) is used to study change in thermal transitions of PS due to sunlight exposure as shown in Figure 10. The changes are as listed in Table 4.

The temperatures of onset (To) peak value (T peak), end set (T e,) Difference in on set – end set (To – Te), heat of enthalpy (ΔH) , height of thermo gram are listed in Table 4. Due to sunlight exposure all the parameters have shifted to low temperatures as expected. The low temperature shift is due to chain cleavages occurred on sunlight exposure [30].

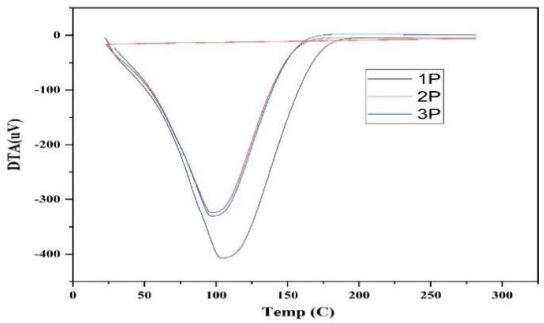

Figure 10: DTA curves of PS exposed to sunlight.

Table 4: Thermal transition temperature properties of sunlight exposed PS.

S.No	Exposure time in hours	T (on set) C	T peak C	T (end set) C	ΔT	ΔH J/g	Height uV

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1	24	57.57	104.12	166.29	108.72	3531	397
2	48	48.95	97.4	148.3	99.35	3401	310
3	72	66.1	98.2	149.83	88.73	3243	316.19

EFFECT OF SUNLIGHT EXPOSURE ON SURFACE MORPHOLOGY OF PS GRANULE

Effcet of sunlight exposure on granular morphology of PS has been investigated using scanning electron microscope (SEM) as shown in Figure 11 a, b & C. . The surface non-irradiated PS granule has a smooth surface but upon exposure to sunlight, the suface become rough. Due io impact of radiation small bores/ pores and cracks appear Granulometrc techniques are applied [32] to estimate pore / crack sixe / density as given in Table 5. With increase of exposure time the pore/ crack size and density have incraesed suggesting destructive action of sunlight radiation.

Table 5: Granular size, granular density and pore size determination from SEM.

S.No	Sunlight exposed In hours	µm/sq mum	1µm- 5µm
1	24	32.90 / spheroid / ellipsoid	10 – 2
2	48	25.27 / spheroid / ellipsoid	12 -15
3	72	27.72 / spheroid / ellipsoid	10 - 10

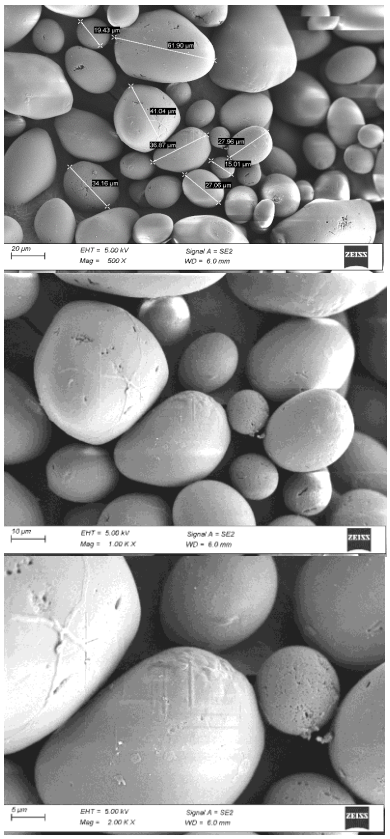


Figure11a: SEM micrographs of PS 24 hours.

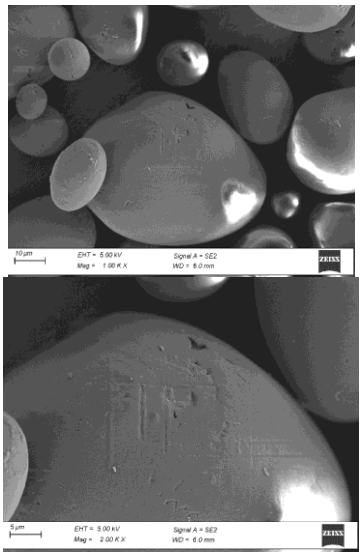
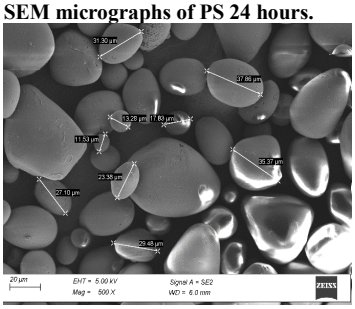
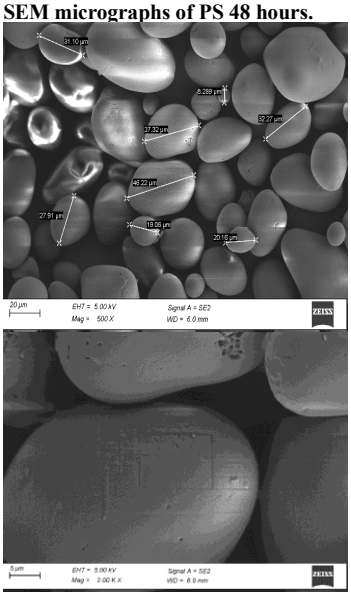


Figure11b: SEM micrographs of PS 48 hours.





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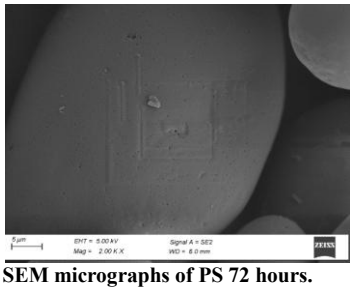

Figure11c: SEM micrographs of PS 72 hours.

HEALTH IMPLICATIONS OF SUNLIGHT EXPOSURE ON POTATO STARCH:

Sunlight exposes of starch has some health concerns/considerations too. Development of green patches on the skin of potato starches under sunlight exposure conditions [34,35]. The green color formation is associated with the presence of glycoalkoloids (GA) and nitrile (N) which have toxic nature and have an impact on human health. Formation of GA&N depend on type/ origin of PS and time of exposes [36,37]. The presence of GA and N can be detected by verifying the following FTIR absorption bands.

S.No	Band	Wave length cm^{-1}
1	O – H, N – H	3340-3300
2	O – H,C – N,C = C	1630-1600
3	C – O	1315-1300
4	C = N	2300-2200

Considering the FTIR spectra of the present studies (Shown in Figure 2) the presence of GA&N is proposed. The intensity of GA and N decreased with sunlight exposure indicate the reduction in concentration of these groups. Further the ESR spectral shape has ill – resolved shapes suggesting the presence of more than one free radical i.e starch free radicals as well as GA and N.

CONCLUSION:

In conclusion sunlight exposure causes changes in chemical structure by the cleavage of chemical groups and molecules leading to the formation of free radicals The free radical formation and chemical structure changes are identified by ESR / FTIR techniques. The XRD data indicate variation of crystalline structure, inter-planar spacing, degree of crystallinity. Williamson - Hall method is used to estimate lattice distortion parameter and crystalline width. Thermal degradation profiles are considerably altered due to sunlight exposure. Activation energy associated with thermal degradation is evaluated to be 60 K J / mole. DTA analysis enables changes in thermal properties of due to sunlight light exposure, Morphology of PS granules has been considerably alter by formation of pores/ bores / cracks due to sun light exposure. Green color development of potato starch skin is associated with formation of glycoalkoloids and nitriles has also proposed by monitoring band position / intensities of FTIR absorption bands.

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