







Development of *Citrus sinensis*-Enriched Superabsorbent Hydrogels for Diaper Applications

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Abstract: Incontinence-associated dermatitis (IAD) is a major concern in preterm infants and elderly patients, often aggravated by prolonged exposure to moisture in diapers. This study presents an eco-friendly superabsorbent hydrogel (OPH5) formulated using *Citrus sinensis* peel powder, sodium polyacrylate, and chitosan. Comprehensive physicochemical analyses revealed an amorphous, porous matrix with high water affinity. OPH5 demonstrated superior fluid absorption (39.08%) and retention (58.4%) under simulated diaper loading conditions. The presence of pectin and cellulose in the *Citrus sinensis* peel powder provides the superabsorbent framework by increasing the surface area of absorption within the superabsorbent hydrogel. *In vitro* skin irritation assays (OECD TG 439) confirmed anti-irritant effect across all tested concentrations, with >70% cell viability compared to control ($p < 0.01$). Limitations of this study include the comparative analysis with synthetic analogues, since it focused on the design of a dry hydrogel from a natural source. Biodegradability analysis and performance testing with marketed preparations can be considered as the future directions of this research.

Keywords: superabsorbent; dry hydrogel; *Citrus sinensis*; orange peel; eco-friendly.

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1. Introduction

Single-use diapers are a major product for parents, since cloth diapers require intensive cleaning to prevent microbial infections in children. Moreover, single-use or disposable diapers fall under the category of Absorbent Hygiene Products (AHP), including menstrual pads and adult incontinence products [1]. It should be noticed that babies are not the only users of these diapers since they also constitute the people affected with mobility, mental impairment, and urinary incontinence. Besides, older adults also employ diapers for urinary incontinence [2,3].

Urinary incontinence is strongly associated with poor Quality of Life (QoL); hence, an effective solution with long-term support is essential [4]. In terms of long-term care, geriatric patients often prefer wearing diapers over behavioral techniques, especially prompted (or) timed voiding, which is considered as 'fostering dependence' and 'embarrassing' [5]. Prolonged diaper usage beyond a prescribed period can lead to Incontinence Associated Dermatitis (IAD),

mainly caused by chronic contact of urine with the skin. Hence, there is a need for improving the absorbing ability of the diaper [6]. This ability is achieved through several approaches. Efficient production of a highly permeable top layer that contacts the skin is necessary to prevent wetting [7]. Designing a lightweight diaper using a superabsorbent material is another significant approach that prevents IAD and skin overhydration [8,9]. The use of synthetic chemicals for promoting high absorption can lead to carcinogenic effects, especially in babies [10]. Acrylic acid and ethyl acrylate are some possible human carcinogens [11].

It is evident that adults produce more AHP waste compared to babies [2,12]. Landfill is the most commonly used technique for disposing of AHP waste, yet it is not feasible in countries with high population density [12,13]. Synthetic SAP does not easily biodegrade (0.12-0.24% semi-annually) due to the stable carbon backbone, irrespective of a minor increase in temperature [14,15]. Furthermore, synthetic SAP reduces fertility. Incineration, adopted in high population density areas, has a higher Global Warming Potential (GWP) compared to landfills [16]. These combust incompletely and release toxic by-products, such as dioxanes, which are harmful to both humans and the environment [1,11,17]. This type of plastic does not combust completely due to its complex structure and forms toxic byproducts and greenhouse gases. Even in the case of landfills, the fertility and nature of soil are greatly diminished due to the permanent settling of AHP waste [11,14,18]. Agricultural productivity of the soil will be damaged by the sorption of water by the superabsorbent. Because this will affect water infiltration and aeration of the soil, leading to erosion [19]. Synthetic SAPs, which are not known for biodegradation, can pose a threat to soil fertility and productivity by acting as a 'microplastic' [20]. Natural SAP exhibits high biodegradability, readily decomposed by microbes even while providing good mechanical strength [7,17].

Incorporating orange peel into a superabsorbent hydrogel formulation will enhance its moisture absorption capacity and biodegradability, providing an effective, eco-friendly alternative to synthetic SAP used in diapers. Because pectin, a polysaccharide, is responsible for the superabsorbent property of the orange peel [21]. Besides, the biodegradability of pectin present in the natural source brings up a new hope in eco-friendly management in AHP [22]. *Citrus sinensis* is rich in flavonoids, which will assist in crosslinking of the SAP, aiding in good retention of moisture by providing mechanical strength of the SAP formulation [23,24]. The organic acids, like citric acid, assist in the crosslinking process of SAP formulation and act as a preservative [24]. This research work focuses on the production and application of a superabsorbent gel for effectively absorbing moisture, which can be used as a suitable superabsorbent for diapers. It does not use synthetic crosslinking agents like glutaraldehyde. This research also specifically focuses on osmotically pressure-driven swelling to maintain super-absorbency. This was achieved by fine-tuning the pH of the formulation [25]. The prepared formulations were subjected to a series of tests evaluating the performance as a superabsorbent gel.

2. Materials and Methods

2.1. Materials.

Chitosan (medium molecular weight, 90%) and Tween 80 were obtained from Sisco Research Laboratories Pvt. Ltd. Anhydrous citric acid was procured from Thermo Fisher Scientific. Benzoic acid, ammonium persulfate, and potassium bromide (all ACS grade) were

purchased from Pallav Chemicals. Silica gel (Elysian blue, 12-20 mm) for moisture control during storage was supplied by Waltzer India.

2.2. Collection and authentication.

The peels of *Citrus sinensis* were authenticated by a certified botanist in the ABS botanical gardens, Salem.

2.3. Preparation of orange peel powder.

Orange peel powder was prepared according to Al-Mhyawi *et al.* [26] with slight modifications. The fruits were washed with distilled water, and the peels were removed from the fruits. The outer layer of the peels was scraped off to avoid colour overlap during the adsorption measurements. Inner peel was cut into fine pieces, ground, and heated at 45 to 60°C in a hot air oven (HEICO). Finally, it was passed through sieve number 175 (0.004 mm) [26].

2.4. Organoleptic characteristic.

This was performed to understand the quality of the prepared *Citrus sinensis* powder. The orange peel powder and excipients were subjected to a blinded organoleptic screening using 5 healthy volunteers. Persons with respiratory problems and allergies were excluded from the study. The protocols and risks of the organoleptic study were explained to the volunteers before the study, both verbally and in writing, and the study was conducted with their consent to participate. The sensory evaluation of orange peel powder was conducted in an environmentally neutral place (free from external smells and distractions) with a panel of 5 subjects, who assessed the appearance, texture, and odour of the powder [27].

2.5. X-ray diffraction.

The sample was placed in an X-ray diffractometer (Malvern). The sample was analyzed in the range of 5° to 80° by irradiating with CuK α radiation. This analytical technique was used to characterize the crystalline structure of the prepared orange peel powder [28,29]. Because the crystallinity influences the water absorption ability of the prepared powder [30].

2.6. Dynamic light scattering.

Particle size distribution was performed by the principle of Dynamic Light Scattering (DLS) using a Zetasizer (Malvern v2.2). The sample was dispersed in water and analyzed with a count rate of 273.6 kcps at 25°C for 10 seconds. [31] This is performed to understand the particle size of the powder, because a smaller particle size leads to a huge surface area, thereby leading to rapid water absorption [32].

2.7. Scanning electron microscopy.

The samples were lyophilized using a freeze dryer (Esquire Biotech EBT10N) and stored in a desiccator containing silica gel as desiccant. The sample was placed under a Scanning Electron Microscope (Carl Zeiss GmbH EVO18). The image was focused with a resolution of 3 nm and Electron High Tension (EHT) of 10 kV under high vacuum. The images were captured at different magnification levels (1,2,3,5,10 μ m). This analysis was performed to understand the surface morphology of the particles of API [33-35].

2.8. FT-IR spectroscopy.

The samples were prepared by the potassium bromide (KBr) pelletization technique by using a hydraulic press (Athena Technologies). The sample pellets were placed in the FT-IR spectrometer (Shimadzu IR Affinity Model 1S) and analyzed in the range of 400 cm^{-1} to 4000 cm^{-1} . This was performed to analyze chemical integrity and drug-excipient interaction before hydrogel formulation. Dry air purging was performed as a moisture control protocol [31,36-38].

2.9. Formulation of orange peel powder hydrogel.

Superabsorbent hydrogels were synthesized via free-radical-initiated solution-phase graft copolymerization, using APS as the initiator and sodium polyacrylate as the crosslinker. Chitosan solution was prepared by dissolving the prescribed quantity (1-2 g) in 1% acetic acid solution. Benzoic acid and Tween 80 were added to this solution. Citric acid was added to this solution to adjust the pH within the range of 5.0 to 6.5. Ammonium persulfate was added as a gel-forming initiator. This was slowly combined with the orange peel mixture, which was already premixed with sodium polyacrylate at a crosslinking ratio of 0.01-0.03, as shown in Table S1. The resultant mixture was thoroughly mixed using a mechanical stirrer at 100 RPM (LabIndia). Finally, the dry superabsorbent hydrogel beads were stored in an airtight container for further analysis. Detailed formulation details are tabulated in Table 1.

Table 1. Formulation table for *Citrus sinensis* loaded superabsorbent hydrogel.

Components	Role	OPH1	OPH2	OPH3	OPH4	OPH5
Chitosan (g)	Monomer	1.0	1.0	1.50	2.0	2.0
Orange peel powder (g)	Superabsorbent	2.0	3.0	1.50	1.50	2.0
Sodium polyacrylate (g)	Polymer	2.0	2.0	3.0	3.0	5.0
Benzoic acid (g)	Preservative	-	-	-	0.05	0.05
Tween 80 (g)	Solubilizer	0.05	-	-	0.1	0.1
Citric acid (mg)	pH adjuster	-	80	-	50	50
Ammonium persulfate (g)	Initiator	0.1	0.05	0.05	0.1	0.1

The *Citrus sinensis* peel contains organic acids and flavonoids capable of crosslinking and forming a superabsorbent gel. The organic acid, like citric acid, present in *C. sinensis* peels, also works as a preservative. However, OPH4 and OPH5 were added with preservatives and additional crosslinking agents to understand the externally added agents on the effect of the superabsorbent property of the SAP hydrogel. The OPH4 and OPH5 were considered to include all excipients, out of which OPH4 and OPH5 were differentiated with the concentration of polymer [24,39].

2.10. Physical appearance.

Properties of the prepared hydrogel were observed to estimate the physical integrity roughly.

2.11. pH.

The sample was analyzed using a digital pH meter (Systronics μ -pH system 362) after prior calibration using the triple-point method. This evaluation was performed to understand the compatibility of the hydrogel with the biological pH [40,41].

2.12. Absorption capacity.

Dry hydrogel particles were initially weighed and transferred to a beaker filled with 0.9% NaCl solution. After a specific period, the swollen hydrogel particles are weighed, and the absorbance capacity is calculated [42,43].

2.13. Leakage test.

It was estimated using the principle of centrifugation. This test evaluates the ability of the hydrogel to retain absorbed liquid under pressure. The sample was centrifuged for 10 minutes at 1000 RPM using a laboratory centrifuge (Medsor Impex MICF2). The decrease in weight of the hydrogel expresses the amount of leakage under pressure [44].

2.14. *In vitro* skin irritation test.

This *in vitro* activity test was performed for the optimized superabsorbent gel in accordance with OECD guidelines TG 439. Phosphate Buffer Saline and 5% sodium dodecyl sulphate (SDS) were used as negative and positive controls, respectively. Since only *in vitro* studies were performed, ethical approval was not required.

2.14.1. Preparation of sample specimens.

Initially, the reconstructed human epidermis (RHE) was incubated at 37°C for 24 hours. The sample (10mg) was added to a small amount of distilled water (5 µL) through a nylon mesh for uniform distribution on the RHE. After a 15-minute exposure at room temperature, phosphate-buffered saline (pH 7.4) was used to rinse the test sample. The RHE model is again incubated for 42 hours at 37°C. This is performed for recovery or further cytotoxic effects.

2.14.2. MTT assay.

The RHE tissues were incubated with 3-(4,5-dimethylthiazol-2yl)-2,5-diphenyltetrazolium bromide (MTT) solution for 3 hours at 37°C. It is then extracted with acidified isopropanol for 4 hours at room temperature. Sodium Dodecyl Sulphate (SDS) and Phosphate Buffer Saline were employed as the positive and negative controls, respectively. The optical density was measured at 570 nm using a UV-Vis spectrophotometer. Cell viability of more than 70% was considered safe and non-cytotoxic in nature. The analysis was performed in triplicate [45-47].

2.15. Stability studies.

Stability testing was performed to observe the physical and chemical integrity of the optimized formulation as per ICH guideline Q1A. The formulation was stored at 40±2°C/75±5% RH over a period of 3 months. The samples were tested on a monthly basis [48,49].

2.16. Statistical analysis.

Statistical analysis was performed using Jamovi 2.3.28. Two-way factor ANOVA with replication was performed for certain evaluation tests, including Absorbency test and Retention ability test, due to the presence of multiple water quantity levels for each formulation (OPH1-OPH5). Tukey's HSD test was conducted as a post-hoc test for ANOVA. One factor ANOVA

was performed for the *in vitro* skin irritation assay and pH, followed by Tukey's HSD test. The number of replicates per test was three.

3. Results and Discussion

3.1. Organoleptic characteristic.

The organoleptic properties of the orange peel powder were qualitatively estimated. The orange peel powder was observed to be light yellow in colour with a strong, tangy odor. The taste was slightly sour. The details of the organoleptic characteristics provided to the subjects are shown in Table S2. This highlights the quality of the *Citrus sinensis* peel powder.

This research study deals with the formulation of a natural superabsorbent hydrogel using *Citrus sinensis* (Orange) peel powder. The organoleptic properties were consistent with the existing literature; however, the bitterness level of orange peel powder reported by Singh et al. [50] was higher than that observed in our research [50]. This may be due to differences in geographical location and ripening, as the ripening process can strongly influence fruit flavour [51]. Bitterness and sourness of the orange peel powder can influence the formation of an interpenetrated polymeric network (IPN). Because naringenin and malic acid are highly responsible for the bitterness and sourness in citrus fruits [52,53]. Malic acid improves crosslinking with chitosan, while naringenin facilitates IPN formation by reacting with the crosslinker via its phenolic hydroxyl groups [54,55]. The sweetness of the orange is not related to good IPN formation. Even though fructose acts as a crosslinking agent, it reduces the quality of the product since it supports Maillard's reaction. This is not confined to fructose; however, it extends to all reducing sugars like glucose, etc. [56] Besides, high concentrations of fructose are required to achieve a stable hydrogel. Low concentrations of Malic acid, on the other hand, provide a stable hydrogel without affecting breaking tenacity [57,58].

3.2. X-ray diffraction.

Analysis revealed the presence of two strong (16.26° , 21.56°) and two feeble peaks (34.72° , 64.1°). The presence of sharp peaks indicates the crystalline nature of the sample. A sharp peak at 21.56° clearly indicates the presence of a specific phytoconstituent, which could serve as a useful superabsorbent in this research. The XRD graph is shown in Figure 1.

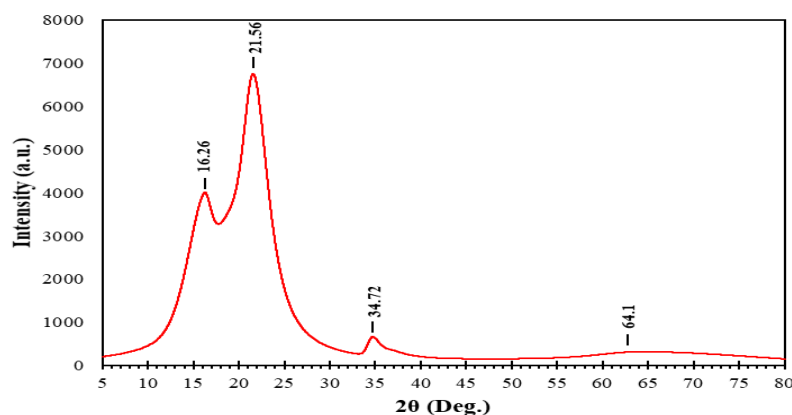


Figure 1. X-ray diffraction graph of *Citrus sinensis* peel extract.

The crystallinity of orange peel Powder was observed to be semi-crystalline, contrary to the existing literature. Interestingly, a faint peak at 64.1° obtained in our research was not mentioned in the existing literature. However, the broad 'almost flat' humps beyond 40°

indicate the amorphous nature of the semi-crystalline powder. The crystalline nature of the powder was confirmed based on the intensity.

Praipipat *et al.* [59] exhibited a characteristic peak at 22.12° (<1450 cps), which was less than our characteristic peak (6757.801 cps) [59]. The presence of pectin in orange peel powder was confirmed by the sharp peak at 21.56°, which is close to the distinct peak mentioned by Yaradoddi *et al.* [60]. This literature also suggests the crystalline part of the semi-crystalline sample [60].

This semi-crystalline feature is essential for an SAP because the crystalline part provides mechanical strength, while the amorphous part provides a high surface area for moisture absorption [61].

3.3. Dynamic light scattering.

The particle size of the sample was mostly in the range of 51.95 nm (Figure 2).

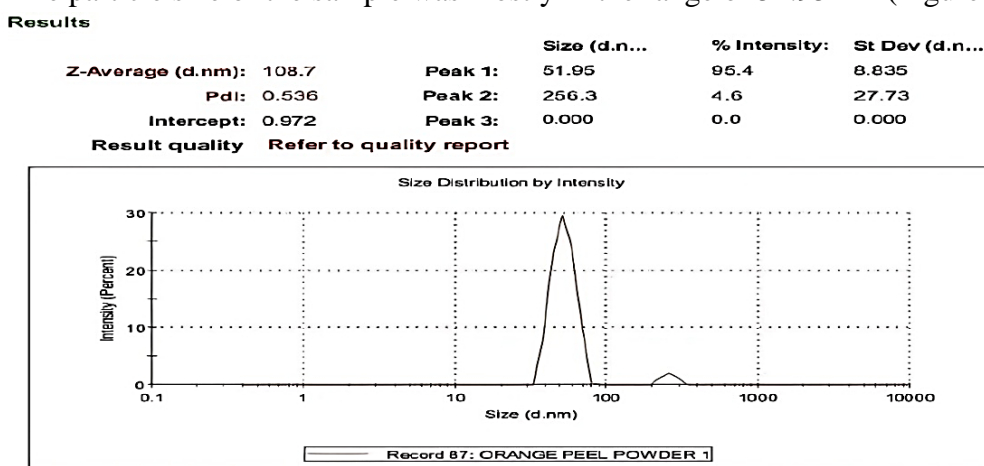


Figure 2. Particle size distribution of *Citrus sinensis* peel powder.

This indicates the efficiency of the preparation process in the size reduction of orange peel powder. However, there were trace amounts of particles falling in the range of 256.3 nm with a feeble intensity (4.6%). The success of the preparation technique was indicated by the minimal standard deviation (± 8.835) in the strongest signal (51.95 nm). The polydispersity Index (PDI) was calculated to be 0.536. The consistency of the process is to be considered due to $PDI > 0.5$.

Fascinatingly, we obtained orange peel powder in nano size (50-270nm), which was confirmed under dynamic light scattering. Hence, the resultant powder can be considered nanoparticles due to its small particle size, as 50% of the sample falls below 100 nm [62].

Generally, milling is considered a top-down approach for nanoparticles under mechanical synthesis. However, sieving can be highly efficient in producing nanostructures, subject to the size of the sieve used. In this research, sieve size (No. 325) was highly influential in nanosizing the orange peel powder [63,64]. Usually, molecular sieves are employed for the preparation of such nanoparticles. It is interesting to note that molecular sieves were not employed, yet the particle size remained below 300 nm. Due to a lack of proper molecular sieves, there was a particle size gap between 51 nm and 250 nm [65].

3.4. Scanning electron microscopy.

Aggregation of nano-sized particles of orange peel powder was observed. Besides, the presence of fibrous components was visible at lower magnifications (5 μ m, 10 μ m), which was

recognized by the presence of voids and cracks. The porous structures were further confirmed with higher magnification. There was a presence of some uneven edges in the porous matrix. The surface morphology of *Citrus sinensis* peel powder was in accordance with the X-ray diffraction analysis of orange peel powder. The microscopic images are shown in Figure 3.

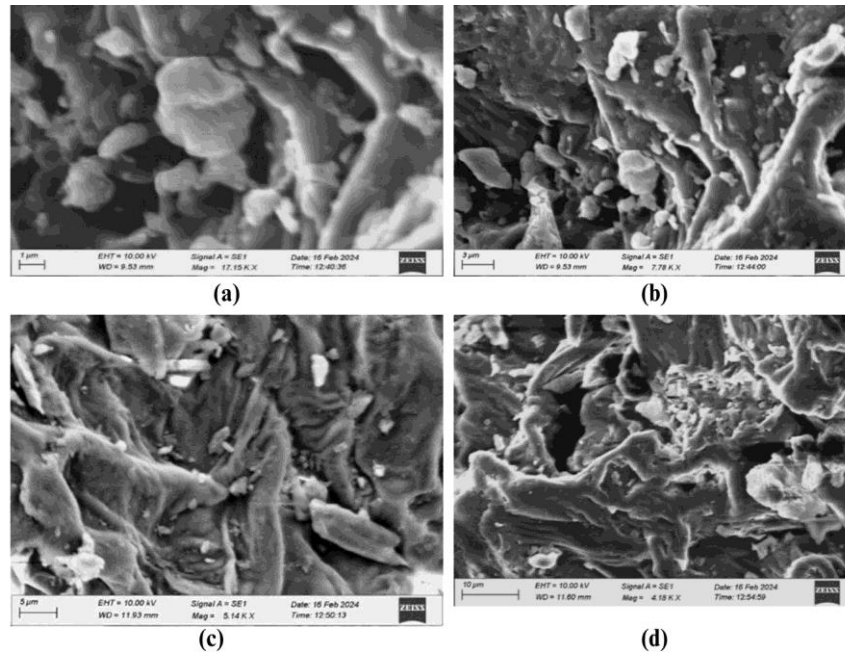


Figure 3. Scanning Electron Microscopy (SEM) images of *Citrus sinensis* peel powder at (a) 1 μm; (b) 3 μm; (c) 5 μm; (d) 10 μm.

The porous meshwork was due to the presence of pectin. Besides, the lack of highly irregular pores may be due to the effective application of heat at 45-60°C [66]. The crystalline nature and aggregation were also confirmed based on SEM images of existing literature [67,68]. Degradation can be due to the presence of certain polysaccharides, which are highly essential for the superabsorbent property. Such substances reduce the electrostatic repulsion [69,70].

3.5. FT-IR spectroscopy.

The presence of polysaccharides was confirmed by the distinct peak at (3333.10 cm⁻¹) and multiple peaks between the range 1030-1102 cm⁻¹ (1054.13 cm⁻¹, 1070.53 cm⁻¹, 1101.39cm⁻¹). Besides, polyphenols may also be present due to the presence of aromatic ring (1608.69cm⁻¹). Presence of aromatic ether was confirmed with the distinct peak observed at 1241.23 cm⁻¹. The FT-IR spectrum of *Citrus sinensis* peel powder is shown in Figure 4.

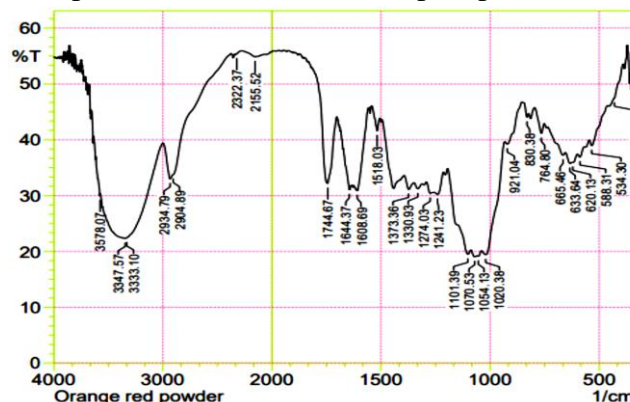


Figure 4. FT-IR spectrum of *Citrus sinensis* peel powder.

The interpretation of the FT-IR peaks is provided in Table 2.

Table 2. Interpretation of Significant peaks of *Citrus sinensis* peel powder.

S. No	Peak (cm ⁻¹)	Interpretation
1	1241.23	C-O stretching
2	1518.03	C=C stretching
3	1744.67	C=O stretching
4	2904.89	C-H stretching
5	3333.10	O-H stretching

The polysaccharides were confirmed by the presence of multiple peaks between 1010-1108 cm⁻¹, attributed to the -CH₂OH group [71]. Especially, the structure of pectin was confirmed by the C=O bond of the methyl ester group (1744.67 cm⁻¹), asymmetric C=O stretching vibration of carboxylic acid group (-COOH), and O-H stretching (333.10 cm⁻¹) [72,73]. The presence of pectin is responsible for the superabsorbent property, owing to the microporous structure. This provides a large surface area to absorb moisture. Besides, pectin is a potential contributor to the effective formulation of this superabsorbent gel, as it acts as a backbone during free-radical polymerization [21,74]. Cellulose was also confirmed by C-H stretching (2904.89 cm⁻¹) and deformation of the C-H bond (1373.36 cm⁻¹) [75].

Cellulose is another important natural SAP that, when combined with pectin, produces synergistic superabsorbent properties [76]. Presence of hesperidin and narirutin can be characterized by the presence of C=O vibration [77]. Hesperidin and narirutin, flavonoids found in citrus peels, are primarily known for their bioactive antioxidant properties and are not reported to have intrinsic superabsorbent characteristics. However, they may contribute to the functional properties or stability of pectin-based hydrogels when present as natural additives or co-extracted compounds [78].

3.6. Physical appearance.

The physical appearance of the prepared hydrogel was dry with minimal moisture due to the presence of solvent (Figure 5). The hydrogel resembled the colour and odour of the orange peel powder.



Figure 5. Dry superabsorbent hydrogel containing *Citrus Sinensis* peel powder.

The physical nature of the prepared superabsorbent hydrogel was dry, corresponding with the existing literature. The initial dry nature is highly necessary for preventing moisture-associated illnesses, like incontinence-associated dermatitis [79,80].

3.7. pH.

All the formulations exhibited pH within the range of 5.5-7 (Figure 6), which is considered the ideal skin pH range. Even though there was a moderately positive correlation ($R^2=0.771$) between Chitosan concentration (%) and pH in the regression analysis, it was not

statistically significant in the one-factor ANOVA test ($p > 0.05$). The regression analysis is tabulated in Table 3. It should be noted that pH values were statistically significant ($p < 0.01$) between different groups (OPH1-OPH5) in one-factor ANOVA.

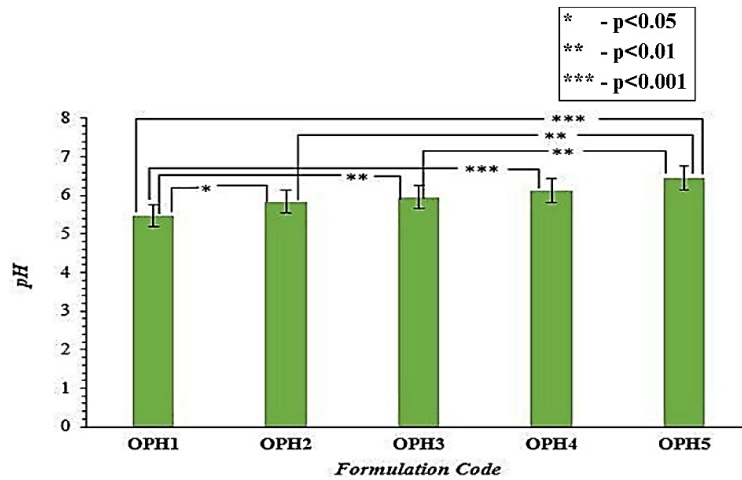


Figure 6. pH of *Citrus sinensis* peel superabsorbent hydrogel formulations (OPH1-OPH5) with Tukey’s test significance, Mean±SD (n=3).

OPH1 was significantly different ($p < 0.05$) from all the formulations. OPH5 was statistically different ($p < 0.01$) from all formulations except OPH4. This is due to the similarity in the formulation design between OPH4 and OPH5, except for the concentration.

Table 3. Regression analysis of Chitosan concentration and pH of the formulation

Title 1	Title 2	Title 3	95% Confidence interval		t	p
			Lower	Upper		
Intercept ^a	5.655	0.173	4.911	6.40	32.70	< .001
Chitosan Concentration (%)						
1.5 – 1.0	0.305	0.300	-0.984	1.59	1.02	0.416
2.0 – 1.0	0.635	0.245	-0.417	1.69	2.60	0.122

The pH of the prepared hydrogel was within the ideal skin pH range (5.5-7). Even though there is a statistically significant increase in pH with constant diaper wear (+0.2), it remains within the ideal pH range, suggesting long-term use [81,82].

3.8. Absorption capacity.

The percentage of liquid absorption by the superabsorbent hydrogel decreased with increasing cumulative liquid volume. All formulations (OPH1-OPH5) exhibited initial absorption greater than 75% (Table 4).

Table 4. Absorption capacity of *Citrus sinensis* peel hydrogel formulations (OPH1-OPH5).

Formulation code	Amount of liquid (mL)	Water absorbed (%)	Absorption capacity	% weight increase	Normalized absorption capacity (g/g)
OPH1	50	77.29±0.48 ^a	27.22±0.75	440.25	3.40±0.09 ^a
	100	58.18±0.01 ^c	48.99±1.03	239.09	1.39±0.00 ^b
	200	45.50±0.01 ^c	70.31±1.50	183.49	0.84±0.00 ^b
	300	18.64±0.01 ^a	35.4±0.77	122.91	0.23±0.00 ^a
OPH2	50	84.66±0.18 ^b	44.14±0.60	651.75	5.52±0.08 ^b
	100	71.39±0.19 ^c	130.08±1.59	349.48	2.49±0.02 ^c
	200	41.65±0.43 ^b	130.04±1.87	171.36	0.71±0.01 ^a
	300	20.39±0.43 ^b	79.98±1.69	125.61	0.26±0.01 ^a
OPH3	50	86.26±0.22 ^b	50.22±0.92	727.75	6.28±0.11 ^d
	100	51.98±0.23 ^b	63.02±0.46	208.25	1.08±0.01 ^a
	200	35.81±0.68 ^a	67.63±1.98	155.78	0.56±0.02 ^a
	300	24.25±1.31 ^c	78.22±4.65	132.01	0.32±0.02 ^a
OPH4	50	85.83±0.39 ^b	48.44±1.55	705.50	6.05±0.19 ^c

Formulation code	Amount of liquid (mL)	Water absorbed (%)	Absorption capacity	% weight increase	Normalized absorption capacity (g/g)
	100	60.02±0.55 ^d	84.73±0.39	250.12	1.50±0.03 ^b
	200	46.64±0.63 ^c	123.38±1.43	187.40	0.87±0.02 ^b
	300	34.05±0.31 ^d	136.59±1.67	151.63	0.52±0.01 ^b
OPH5	50	86.78±0.44 ^c	52.53±2.01	756.63	6.57±0.25 ^e
	100	50.05±0.33 ^a	60.64±1.21	200.18	1.00±0.01 ^a
	200	58.13±2.30 ^d	168.2±11.40	238.81	1.39±0.13 ^c
	300	39.08±0.03 ^d	185.63±5.52	164.15	0.64±0.00 ^b

Mean±SD (n=3); Superscript letters (a-h) indicate statistical differences. Values with different letters are significantly different (p<0.05) within the same water quantity.

The Normalized Absorption Capacity (NAC) of hydrogel was calculated within the range between 0.2 g/g and 6.6 g/g. This was in accordance with Al-Mhyawi *et al.* [26], who exhibited NAC of 1.9g/g [26].

However, the retention of this property was observed under high liquid load in OPH5 (39.08%) even after the cumulative liquid load of 650 mL. It was due to the presence of a polymer (Sodium polyacrylate), which retarded the depletion of the superabsorbent hydrogel's absorbent properties. The results revealed that the hydrogel absorbency is significantly influenced by both the formulation composition and the volume of water applied, as indicated by the highly significant ANOVA outcomes (p<0.01). Tukey's HSD test revealed that at 50 mL, OPH3, OPH4, and OPH5 exhibited statistically similar (non-significant with p>0.05) absorbency, forming the top-performing cluster. Lower-volume groups (e.g., (OPH1: 300 mL) and (OPH2: 300 mL)) differed significantly from higher-performing groups, indicating volume-dependent absorbance limitations in those formulations. OPH5 retained performance at higher volumes better than others, showing promise for extended-use applications. The absorbance capacity in relation to the amount of water added is graphically represented in Figure 7.

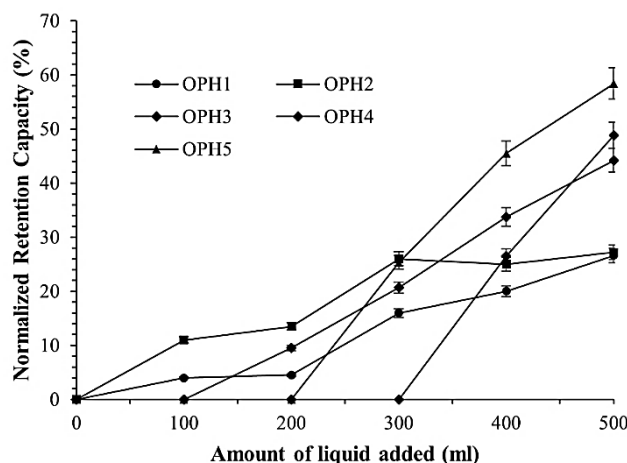


Figure 7. Retention capacity (%) of *Citrus sinensis* peel powder hydrogel formulations (OPH1-OPH5).

The prepared formulations (OPH1-OPH5) proved to be excellent superabsorbent hydrogels, since they absorbed more than 100% of their initial weight even after a cumulative liquid addition of 650 mL [83]. The depletion in absorbency with increased water addition is due to the gradual weakening of the mechanical strength of the crosslinked polymeric network [84]. The presence of hydrophilic phytoconstituents can enhance the absorbency nature of superabsorbent hydrogel [85]. It also aids effective retention due to the dominant hydrophilic interaction between water molecules and the SAP hydrophilic meshwork [86]. Peels of *Citrus sinensis* are known for a lot of hydrophilic components, including ascorbic acid, citric acid, and pectin [87,88]. The electric charges of the polymers in the superabsorbent hydrogel played

a crucial role in liquid retention through a semi-interpenetrating polymeric network (SIPN). This shows better mechanical properties than a full interpenetrating network (IPN) [89,90]. *Citrus sinensis* powder is a rich source of carboxyl and hydroxyl groups, providing organic acids (Ascorbic acid, citric acid) and pectin, which enhance crosslinking ability. [91,92] Interestingly, the weakly acidic nature of the superabsorbent polymer is essential for its promising water absorption nature. pH generally has an influence on electric charge density, through which the osmotic pressure can be maintained. The absorption capacity can be increased by maintaining the osmotic pressure. Moreover, the initial acidic pH helps in the absorption of high moisture content. The absorbance of all SAP hydrogel formulations (OPH1-OPH5) decreased with increasing liquid volume, likely due to a shift toward neutral pH. Furthermore, small particle size and large surface area were responsible for efficient water absorption capacity in this research [25,93].

3.9. Leakage test.

Retention ability was significantly different among formulations at each water load ($p < 0.01$). OPH5 showed the highest retention at 500 mL (58.4%), while OPH2 and OPH3 retained less. OPH5 displayed substantial retention only at higher volumes (>300 mL). Statistically significant differences are identified by non-overlapping letters in Table 5.

Table 5. Retention ratio of various orange peel hydrogel formulations (OPH1-OPH5).

Formulation code	Liquid added (mL)	Retention liquid (mL)	Retention ratio	Retention ability (%)
OPH1	200	9	0.045	4.49±1.64 ^a
	300	48	0.16	16.01±1.06 ^b
	400	80	0.2	20.00±2.20 ^a
	500	133	0.266	26.59±6.63 ^a
OPH2	200	27	0.135	13.53±4.32 ^b
	300	78	0.26	26.00±6.03 ^b
	400	100	0.25	25.00±2.24 ^a
	500	136	0.272	27.20±4.11 ^a
OPH3	200	19	0.095	9.52±2.21 ^a
	300	62	0.207	20.67±3.85 ^b
	400	135	0.338	33.75±3.81 ^b
	500	221	0.442	44.21±8.94 ^b
OPH4	200	0	0	0.06±0.06 ^a
	300	0	0	0.05±0.04 ^a
	400	106	0.265	26.51±3.98 ^a
	500	244	0.488	48.85±7.38 ^b
OPH5	200	0	0	0.03±0.03 ^a
	300	76	0.253	25.32±5.40 ^b
	400	182	0.455	45.33±4.16 ^c
	500	292	0.584	58.49±3.29 ^c

Mean±SD (n=3); Superscript a,b,c,d Formulations with the same letter at the same water level are not significantly different ($p < 0.05$) under Tukey's HSD test pairwise comparisons.

Increased frequency of diaper changes critically increased the stress levels of pre-term infants. Even though respiratory rate was not highly affected, heart rate was significantly increased during most diaper changes ($\geq 74\%$). Adults often prefer treatments that maintain a dry pelvic environment with minimal effort, such as using disposable diapers that reduce the need for frequent changes [5,94]. The crosslinking ability of the porous network of superabsorbent polymer is required for excellent retention of absorbed liquid. This porous network must be flexible in order to retain the absorbed liquid and must not dissolve in it. The mechanical strength of the SAP improves the retention and must not solely depend on the super-absorbency alone [95,96]. The retention ratio increased with an increase in polymer

concentration. This implies that polymer concentration is crucial for improving retention, regardless of superabsorbent concentration. Polyacrylic acid is known for its excellent liquid retention due to its promising crosslinking ability, in addition to its high water absorption capacity [84,97].

3.10. *In vitro* skin irritation test.

There was no dose-dependent response for the optimized hydrogel (OPH5). The cell viability, along with absorbance observed at 570 nm with an increase in dose, is graphically represented in Figure 8 and Table 6. Within the range of 0.3 µg/mL to 6.53 µg/mL, there was a sharp increase in inhibition of skin irritation, indicating efficacy even at low concentrations. The one-way ANOVA reported that the percentage of inhibition of skin irritation in the MTT assay was statistically significant ($p < 0.001$). Pairwise comparisons under Tukey’s HSD revealed a statistically significant difference ($p < 0.01$) in % inhibition between the control and all doses (Table S3).

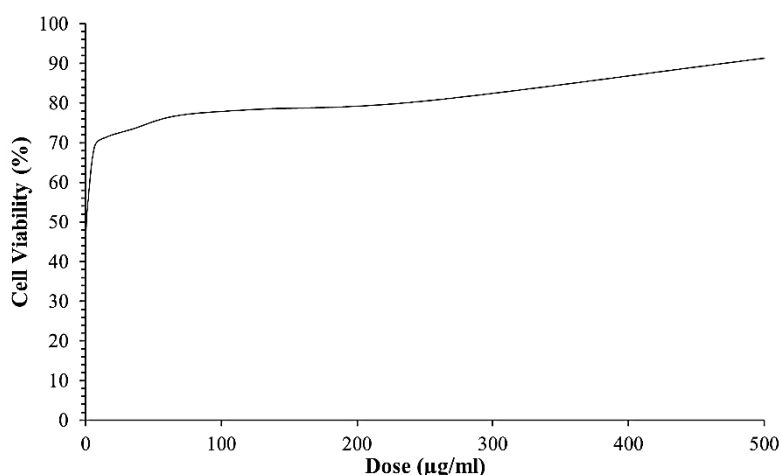


Figure 8. *In vitro* anti-skin irritant activity of optimized orange peel superabsorbent hydrogel (OPH5).

The poor cell viability (<50%) under low concentrations is due to the poor mechanical strength of the SAP hydrogel. Besides, the concentration of trace elements that cause cytotoxicity is not retained at low levels in the product due to the lack of structural integrity of the SAP hydrogel [98,99].

Table 6. Absorbance values at 570 nm and Percent cell viability of optimized superabsorbent hydrogel (OPH5) in the MTT assay.

S. No	Tested sample concentration (µg/mL)	Trial 1		Trial 2		Trial 3		Mean cell viability
		OD Value	% cell viability	OD Value	% cell viability	OD Value	% cell viability	
1	Control	0.800	96.02	0.790	97.99	0.810	96.68	96.90±1.00 ^a
2	500	0.650	90.99	0.630	91.64	0.645	91.14	91.26±2.05 ^b
3	250	0.580	80.56	0.560	80.56	0.575	80.29	80.53±2.20 ^b
4	129	0.790	78.60	0.770	77.50	0.780	78.95	78.47±1.21 ^c
5	67.5	0.750	76.65	0.735	77.29	0.740	76.43	76.88±2.47 ^{cd}
6	34.6	0.690	73.91	0.680	71.28	0.695	75.08	74.70±1.94 ^{de}
7	15.4	0.665	69.06	0.650	73.98	0.660	71.15	72.19±0.44 ^{ef}
8	6.53	0.540	67.87	0.550	70.25	0.555	68.69	69.85±0.76 ^{fg}
9	1.3	0.400	55.67	0.410	52.91	0.405	51.32	53.41±0.16 ^g
10	0.3	0.350	45.47	0.340	47.12	0.360	49.54	47.98±0.34 ^h

Mean±SD (n=3); Superscript letters (a, b, c, d, e, f, g, h) Formulations with the same letter at the same water level are not significantly different ($p < 0.05$).

The *in vitro* anti-irritant is not a direct therapeutic efficacy of the superabsorbent hydrogel. In fact, the skin irritation is greatly reduced when the saline water contact time is reduced. Besides, skin irritation is the least likely to occur when the hydrogel formulation meets the optimal skin period change. Because, the skin flora is not damaged, it contributes to the safe survival of the skin tissues. The acidic nature of the orange peel powder can be a critical factor for exerting therapeutic efficacy by preventing urease enzyme activation [100,101].

3.11. Stability studies.

A slight decrease in pH of the optimal hydrogel was observed over time during the stability studies (Table 7). This indicates a decrease in the formulation's stability. However, the formulation still stayed within the optimal pH range. Besides, the absorption capacity was excellent even after the prescribed period [102].

Table 7. Stability studies of optimized orange peel superabsorbent hydrogel (OPH5).

S. No	Tests	Initial	Month-1	Month-2	Month-3
1	Appearance	No change	No change	No change	No change
2	Odour	No change	No change	No change	No change
3	pH	6.45±0.15	6.37±0.55	6.20 ±0.20	6.08 ±0.30
4	Anti-irritant activity (%)	91.26±2.05	91.05±1.80	90.89±2.32	91.02±2.37

4. Conclusions

This study successfully formulated a superabsorbent hydrogel using *Citrus sinensis* (orange) peel powder as the active ingredient, which exhibited excellent absorbency due to its nearly amorphous structure. Incorporation of a crosslinking agent significantly improved liquid retention, with the optimized formulation (OPH5) demonstrating favorable skin compatibility, making it a promising candidate for Absorbent Hygiene Products such as diapers.

However, the study is limited by the absence of benchmarking against commercial superabsorbent polymers, a lack of biodegradability data under real disposal conditions, and limited mechanical stability assessments related to prolonged use. Addressing these gaps through comprehensive biodegradation studies, mechanical and fatigue testing, and comparative analyses will be essential to validate the hydrogel's environmental impact and commercial viability.

Future research should focus on these aspects, alongside long-term performance in practical settings and the exploration of functionalities, such as antimicrobial incorporation, to broaden clinical applications. These efforts will support the effective commercialization of *Citrus sinensis*-based superabsorbent hydrogels, potentially providing an eco-friendly alternative to synthetic polymers in the hygiene sector.

Author Contributions

Conceptualization, M.C.R.; methodology, S.G.; software, R.S.; validation, N.V. and P.C.; formal analysis, N.V. and S.G.; investigation, P.C.; resources, N.V.; data curation, R.S.; writing—original draft preparation, D.A.; writing—review and editing, S.G.; visualization, N.V., D.A.; supervision, M.C.R.; project administration, M.C.R. All authors have read and agreed to the published version of the manuscript.

Institutional Review Board Statement

Not applicable.

Informed Consent Statement

Informed consent was obtained from volunteers for the organoleptic studies.

Data Availability Statement

All data created or analyzed in this study are available in this manuscript and supplementary data. Data sharing is not applicable.

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Conflicts of Interest

The authors report no conflicts of interest. The authors alone are responsible for the content and writing of this article.

Abbreviations

Abbreviation	Definition
ANOVA	Analysis of Variance
APS	Ammonium Persulfate
FTIR	Fourier Transform Infrared Spectroscopy
HSD	Honestly Significant Difference
IAD	Incontinence-Associated Dermatitis
IPN	Interpenetrating Polymeric Network
MTT	3-(4,5-Dimethylthiazol-2-yl)-2,5-diphenyltetrazolium Bromide
NAC	Normalized Absorption Capacity
OD	Optical Density
OECD	Organisation for Economic Co-operation and Development (for Toxicity Studies)
OPH	Orange Peel-based Hydrogel
pH	Potential of Hydrogen
RHE	Reconstituted Human Epidermis
SAP	Superabsorbent Polymer
SEM	Scanning Electron Microscopy
UV-Vis	Ultraviolet-Visible
WHO	World Health Organization
XRD	X-Ray Diffraction
QoL	Quality-of-Life

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Supplementary materials

Table S1. Crosslinking ratios of Citrus sinensis peel superabsorbent hydrogel formulations (OPH1-OPH5)

Formulation	Chitosan (g)	Polyacrylate (g)	APS1 (g)	Crosslinking ratio (APS / (chitosan + polyacrylate))
OPH1	1.0	2.0	0.1	0.033
OPH2	1.0	2.0	0.05	0.0167
OPH3	1.5	3.0	0.05	0.0111
OPH4	2.0	3.0	0.1	0.02
OPH5	2.0	5.0	0.1	0.0143

Table S2. Organoleptic results of Citrus sinensis peel powder in the blinded study (n=5).

Formulation	Subject-1	Subject-2	Subject-3	Subject-4	Subject-5
Colour	Yellow	Orange	Slight yellow	Slight yellow	Yellowish white
Odour	Strong	Strong tangy	Strong tangy	Strong	Strong tangy
Taste	Slightly sour	Sour	Bitter	Slight sour	Bitter

Table S3. Pairwise comparisons were conducted by Tukey’s HSD test between different doses and the control for the MTT assay.

Dose (µg/mL)	Parameter	0.30	1.30	6.53	15.40	34.60	67.50	129.00	250.00	500.00	Control
0.30	Mean difference	—	-5.92** ¹	-21.6*** ²	-24.02***	-26.05***	-29.41***	-30.97***	-33.10***	-43.9***	-49.52***
	p-value	—	0.003	<.001	<.001	<.001	<.001	<.001	<.001	<.001	<.001
1.30	Mean difference	N/A ³	—	-15.6***	-18.10***	-20.12***	-23.49***	-25.05***	-27.18***	-38.0***	-43.60***
	p-value	N/A	—	<.001	<.001	<.001	<.001	<.001	<.001	<.001	<.001
6.53	Mean difference	N/A	N/A	—	-2.46	-4.49* ⁴	-7.85***	-9.42***	-11.54***	-22.3***	-27.96***
	p-value	N/A	N/A	—	0.597	0.038	<.001	<.001	<.001	<.001	<.001
15.40	Mean difference	N/A	N/A	N/A	—	-2.03	-5.39**	-6.96***	-9.08***	-19.9***	-25.50***
	p-value	N/A	N/A	N/A	—	0.803	0.008	<.001	<.001	<.001	<.001
34.60	Mean difference	N/A	N/A	N/A	N/A	—	-3.37	-4.93*	-7.05***	-17.8***	-23.47***
	p-value	N/A	N/A	N/A	N/A	—	0.215	0.018	<.001	<.001	<.001
67.50	Mean difference	N/A	N/A	N/A	N/A	N/A	—	-1.56	-3.69	-14.5***	-20.11***
	p-value	N/A	N/A	N/A	N/A	N/A	—	0.947	0.136	<.001	<.001
129.00	Mean difference	N/A	N/A	N/A	N/A	N/A	N/A	—	-2.12	-12.9***	-18.54***
	p-value	N/A	N/A	N/A	N/A	N/A	N/A	—	0.761	<.001	<.001
250.00	Mean difference	N/A	N/A	N/A	N/A	N/A	N/A	N/A	—	-10.8***	-16.42***
	p-value	N/A	N/A	N/A	N/A	N/A	N/A	N/A	—	<.001	<.001
500.00	Mean difference	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	—	-5.64**
	p-value	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	—	0.005
Control	Mean difference	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	—
	p-value	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	—

¹ p<0.01; ² p<0.001; ³ Not applicable; ⁴ p<0.05.