Pickering emulsion polymerization of styrene towards alfa-functionalized polystyrene latex: parameters and process conditions

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Abstract: Polymerization conditions in the Pickering emulsion of colloidal silica-stabilized polystyrene latex were optimized. The influence of process conditions on various parameters, including particle size and distribution was investigated. Monomodal particles distribution was obtained by conducting the process at a temperature of 80°C in a slightly acidic environment (pH = 5), while maintaining fast monomer conversion. It has been shown that the amount of stabilizer used in the polymerization process affects the rate of polymerization, therefore the temperature of the process should be adjusted to the amount of colloidal silica used in the process.

Keywords: Pickering emulsion, polymerization, morphology.

Emulsyjna polimeryzacja styrenu metodą Pickeringa wobec alfa-funkcjonalizowanego lateksu polistyrenowego: parametry i warunki procesu

Abstrakt: Zoptymalizowano warunki polimeryzacji w emulsji Pickeringa lateksów polistyrenowych stabilizowanych krzemionką koloidalną. Zbadano wpływ warunków procesu na m.in wielkość i rozkład cząstek. Monomodalny rozkład cząstek uzyskano prowadząc proces w temperaturze 80°C w środowisku lekko kwaśnym (pH = 5), przy zachowaniu szybkiej konwersji monomeru. Wykazano, że ilość stabilizatora użytego w procesie polimeryzacji wpływa na szybkość polimeryzacji, dlatego też temperatura procesu powinna być dostosowana do ilości używanej w procesie krzemionki koloidalnej.

Słowa kluczowe: emulsja Pickeringa, polimeryzacja, struktura.

Nanotechnology is transforming our lives and the economy. Nanotechnology influences microprocessors, chemicals, and biology, allowing the creation of new products with improved functionality. Pickering emulsions, which use inorganic stabiliser particles to reduce petrochemicals in new products, are becoming more popular in the industry. Traditional emulsifiers break down into chemicals that increase VOC and SVOC concentration, therefore eliminating them can lessen them. Emulsifiers can also irritate or decrease fertility [2–3]. Pickering emulsions, unlike surfactant-stabilized emulsions, are stabilised by solid nanoparticles that can be organic or inorganic and of diverse types [4–13]. Numerous nanoparticle types and shapes can stabilise Pickering emulsion polymerization has affected the research for suitable techniques to synthesise nanocomposites in situ. Maricruz Solís-López *et al.* [14] considered using LUDOX AM, colloidal dispersion of silica nanoparticles with a diameter of 12 nm, as a stabiliser for styrene polymerization with polar monomers 4-vinylpyridine (4VP), sodium vinylbenzene sulfonate (VBS), and poly(ethylene glycol) methyl ether methacrylate (PEGMA). VBS, 4VP, and PEGMA with styrene formed Pickering emulsions, whereas 0.5% VBS and 4VP with or without cetyltrimethylammonium bromide (CTAB) activated by ammonium persulfate (APS) did not. Fouconnier *et al.* [15] did another intriguing Pickering emulsion polymerization of styrene research. APS was the initiator, and commercial product Bindzil 830 which is 10 nm colloidal silica was stabiliser. For comparison, they polymerized CTAB-modified silica nanoparticles (mSP). The polymer particle size increased with ammonium persulfate content. CTAB-modified silicas also produced

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smaller polymer particles than when colloidal silica was used alone. Sang Deuk *et al.* [16] proposed polymerizing styrene Pickering emulsions stabilised by graphene oxide (GO) and triggered by 2,2′-Azobisisobutyronitrile (AIBN) to create polystyrene-GO microspheres. Xuan [17] stabilised Pickering emulsion polymerization with dodecylamine-functionalized graphene quantum dots. Graphene quantum dots were selected because they have better surface activity than graphene and are comparable to organic surfactants. As previously reported, AIBN polymerized styrene to create a latex with homogenous morphology and a limited diameter distribution that might be employed in emulsion polymerization, sensor, catalyst, and energy storage. Xiaomei *et al.* reported another intriguing Pickering emulsion polymerization method. They stabilised a 120-nm polystyrene nanocomposite with cationic-charged self-made titania hydrosol modified by anionic sodium styrene sulfonate. UV light and 48 hours of stirring at ambient temperature polymerized the reaction liquid. In Pickering's emulsion, weak attractive interactions between the oppositely charged stabiliser and initiator ions affect the polymerization.

This study focused on polymerizations with cationic salts of radical initiator AAPH and hydrophobized anionic colloidal silica CC-301 to ensure adequate process kinetics, high final conversion, decomposition modality, and stability.

EXPERYMENTAL PART

Materials

The following raw materials were used in the work: high-purity styrene (Synthos Company, CAS 100-42-5); 2,2′-Azobis(2-methylpropionamidine) dihydrochloride (AAPH) (Sigma – Aldrich, CAS 2997-92-4); colloidal silica CC-301 with a mean particle diameter of 7 nm and CC-401 with a mean particle diameter of 12 nm, (Nouryon, CAS 1239225-81-0); hydrochloric acid (Sigma – Aldrich, CAS 7647-01-0).

Methods

Polymerization procedure

Polystyrene nanocomposites were prepared in a sealed glass vessel using 64 g of styrene and varied amounts of colloidal silica in an aqueous phase. Styrene was mixed with demineralized water, hydrochloric acid, AAPH, and colloidal silica. After 15 minutes of nitrogen bubbling, the liquid was stirred at 300 rpm to disperse the monomer. After heating, the reaction mixtures reached the desired temperature. Polymerization rates were measured during the polymerisation process, samples were taken, and the dry content was assessed, allowing the styrene conversion to be computed. Curves are subsequently displayed and compared from the determined measurement points.

Thermogravimetric analysis (TGA)

For thermogravimetric analysis, the sample was dried. A TGA/DSC thermogravimetric analyser (Mettler Toledo, USA)) was used for measurements. Temperature-dependent mass loss and heat flux change curves were recorded by the sensor. Three temperature stages were used in the experiment. The first step entailed maintaining a temperature of 30°C and a flow rate of 50 ml/min of N_2 gas for 10 minutes. In the second step, the N_2 gas flow rate was maintained at 50 ml/min while the temperature was increased from 30 to 600°C at 10°C/min. In the third and final stage, the temperature was raised from 600 to 1000°C at 10°C/min. To pyrolyze the organic/polymer fraction without oxidation, the thermal breakdown was done at 30–600°C in a nitrogen (N_2) environment. The reaction gas was then converted to air at 600–1000°C to burn non-volatile breakdown leftovers. Thermogravimetry uses discrete mass changes to determine sample composition. Polymers and other organic additives decompose at 600°C. The non-volatile residue (ash) that remains after thermal decomposition is oxidised when the measurement environment is changed from inert to oxidising (air).

Mean particle size

The Beckman Coulter® N4 Plus sub-micron particle size analyser (Beckman Coulter, IN, USA) determines the mean particle size using PN ISO 13321. Granulometric examination of particle size distribution is of interest. Photon correlation spectroscopy in optics measures light intensity fluctuations in a material. Photon Correlation Spectroscopy (PCS) measures particle size. The approach analyses the specimen in liquid suspension without damaging its structure. The suggested approach measures laser light scattering fluctuations on Brownian motion particles.

Particle size distribution

Beckman Coulter's LS 13 320 (Beckman Coulter, IN, USA) measured particle size. The average and spread of particle sizes may be determined using this laser light diffraction analyser. This instrument measures dynamically with modern technologies. The approach allows the specimen to be examined in equilibrium (in a liquid solution) without harming or altering its structure.

UV-VIS spectroscopy

The UV-VIS spectrophotometer Evolution 220 series (Thermo Fisher Scientific, MA, USA) analysed liquid aqueous samples. UV-VIS spectrophotometry uses visible and near-infrared electromagnetic energy from 200 to 1100 nm. Absorption bands with a wavelength-absorbance connection characterise electron oscillation and rotation. Liquid samples were prepared by diluting the solution in water. 4 ml of the solvent used to prepare the solution was introduced into the purified cuvette to record the background. The cuvette was then cleaned, and 4 ml of the test sample was introduced.

Transmission electron microscopy (TEM)

The TEM observations were performed with a JOEL JEM 1011 electron microscope (Joel, Japan) at an accelerating voltage of 100 kV. Liquid samples of latexes have been diluted in water and one drop of the prepared solution has been put on copper mesh coated with carbon.

Design of experiment and data analysis

The study used central composite face-design (CCF) to evaluate two SiO_2 particle sizes (as a block variable with two levels) and four Pickering emulsion polymerization variables. The Combinatorial Complete Factorial (CCF) requires 2k + 2(k-1) + n, centre experimental trials for k variable changes. –1, 0, and +1 signified three hierarchical layers of variables. This strategy uses quantitative or qualitative factors. Table 1 details experimental settings and parameters. Pickering emulsion polymerization temperature, pH, and SiO_2/styrene ratio were examined in this work. The CCF chose 16 tests.

Table 1. The experimental matrix of the central composite design (CCF) with a central point

Parameters/ levels	Polymerisation temperature, °C	pH of water phase	SiO_2/styrene ratio
Low	68	5	20
High	80	7	30
Centre	74	6	25

The original parameter ranges were determined by the properties of the raw ingredients used for polymerisation. The polymerisation process was carried out in an aqueous phase with an acidic pH to avoid precipitation of the cationic initiator from its salt. Furthermore, the reduction of the pH of the aqueous phase was caused by the anionic character of the colloidal silica, as reducing the pH affects its capacity to repel each other in a controlled manner. The colloidal silica type and type were chosen due to the nano stabiliser' smallest feasible particle size and its functionalisation with gamma-glycidoxypropyltrimethoxysilane, which permits this type of raw material to be employed in polymerisation at acidic pH. The colloidal silica to monomer ratio was established based on mathematical assumptions derived from the employed model of covering the bigger polymer particle with 7nm colloidal silica. The temperature conditions, on the other hand, were chosen based on the calculated half-life of the AAPH initiator, so that, on the one hand, the Pickering emulsion polymerisation time was as short as possible and as close to classical emulsion polymerisation processes as possible, and, on the other hand, the decomposition time of a significant portion of the initiator was not shorter than that of styrene polymerisation. This is because processes with factors limiting the polymerisation rate, and hence with slower polymerisation kinetics, resulted in lower conversion rates when higher polymerisation temperatures were applied.

Fig. 1. Opacity of water phase based on AAPH with CC-301 colloidal silica (left 0% AAPH, right 2% AAPH)

A multivariate analysis of variance (ANOVA) was used in this study to find variables that statistically affect product quality. By using this method, any independent variable value has the same effect on the dependent variable. One variable significantly affects the other, according to the alternative hypothesis. All levels of the dependent variable are affected equally by the independent variable. Hypotheses are related to each independent variable. Additionally, the independent factors' joint effect on the continuous dependent variable is called the interaction. To determine if the parameter being evaluated has a local or global maximum, the methodology uses information from the planned experimental design.

RESULTS AND DISCUSSION

Anionic colloidal silica solution after the addition of cationic salt as a radical initiator

Considering the ionic nature of the initiator and oppositely charged-colloidal silica, a preliminary study exam-

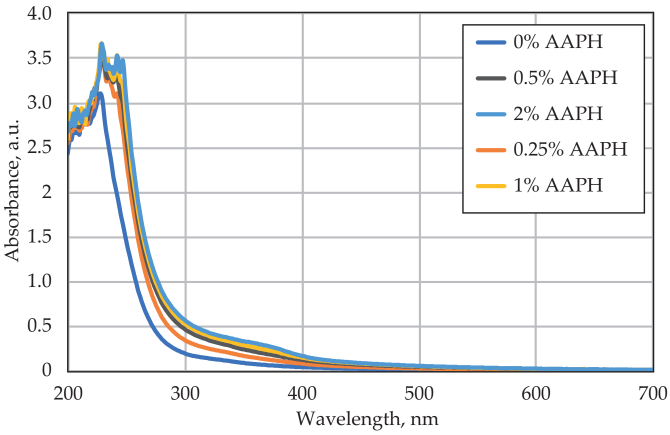

Fig. 2. Absorbance of AAPH/CC-301 solutions

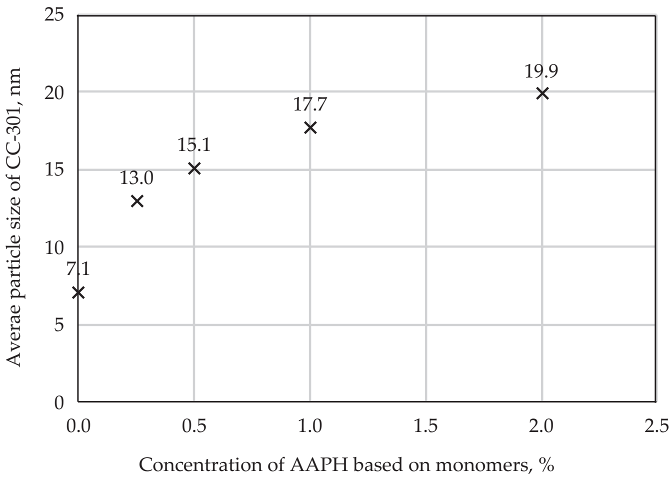

Fig. 3. Average particle size of CC-301 upon addition of a different amount of AAPH initiator

ined how initiator quantity affects the aggregation of colloidal particles. The investigation observed modest turbidity shortly after adding the initiator (Fig. 1). The study measured the mean particle diameter of mixtures with increasing AAPH concentrations using UV-VIS.

Figure 2 shows the positive relationship between AAPH concentration and absorbance, and Figure 3 shows the increase in the mean particle size of the colloidal silica. The increase in mean particles indicates the initiator's proximity to the colloidal silica surface, which stabilises the growing particles during polymerization in Pickering emulsion. The observed effect of AAPH on colloidal silica diameter suggests that Pickering emulsion is stabilised by partial destabilisation of colloids before the reaction. The observed two- or three-fold increase in colloidal silica diameter suggests the existence of agglomerates before AAPH-triggered polymerization. It leads to the conclusion that in certain conditions polymer particle could be stabilized during the process by bigger nanoparticles than it was used prior to the addition of AAPH.

Aging test of CC 301 colloidal silica in slightly acidic pH

Based on particle and absorbance results, the study aged polymers at neutral and weakly acidic (pH 5) conditions for the average polymerization process. This was done to confirm that weak interaction-induced agglomeration is not part of a larger destabilisation process. For identical samples under process conditions, the mean diameter was measured over 8 hours to reach this goal. Analysis shows no precipitation or particle size changes. The raised average particle size system showed stability across the 8-hour testing, which is sufficient for most procedures.

Matrix-based data analysis

Based on the results of the AAPH investigation on colloidal silica, an experimental design was created to manufacture polystyrene latex with a certain shape for the future synthesis of styrene copolymers in Pickering emulsion. Colloidal silica particle size, aqueous phase pH, and process temperature affect polystyrene latex polymerization kinetics in Pickering emulsion. The latexes were morphologically examined. The latexes were characterised by average particle size and dispersion. ANOVA analysis followed.

Figure 4 shows that decreasing colloidal silica concentration from 30 to 20% per monomer increased particle size distribution mode. Insufficient colloidal silica coverage may have led polymer particles to agglomerate. The aggregation resulted in an agglomerated latex at the emulsion polymerization stage, which was characterized by accelerated sedimentation during storage after polym-

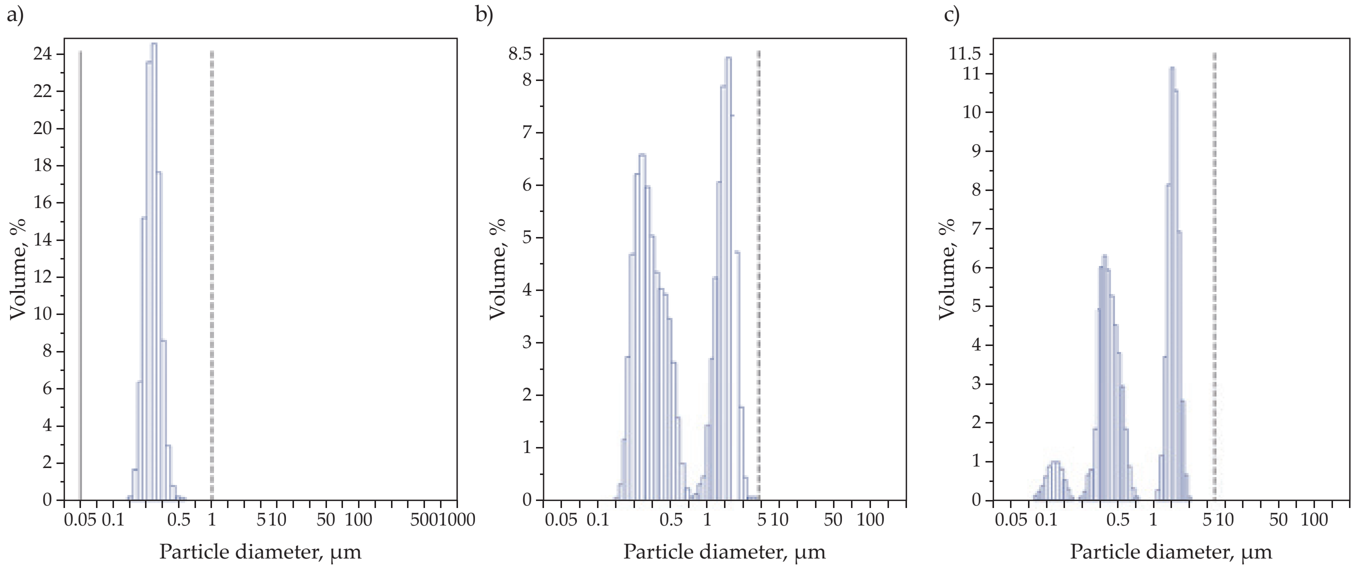

Fig. 4. Particle size distribution: a) polystyrene latex with 30% colloidal silica polymerized in pH 5, b) polystyrene latex with 20% colloidal silica polymerized in pH 5, c) polystyrene latex with 20% colloidal silica polymerized in pH 7

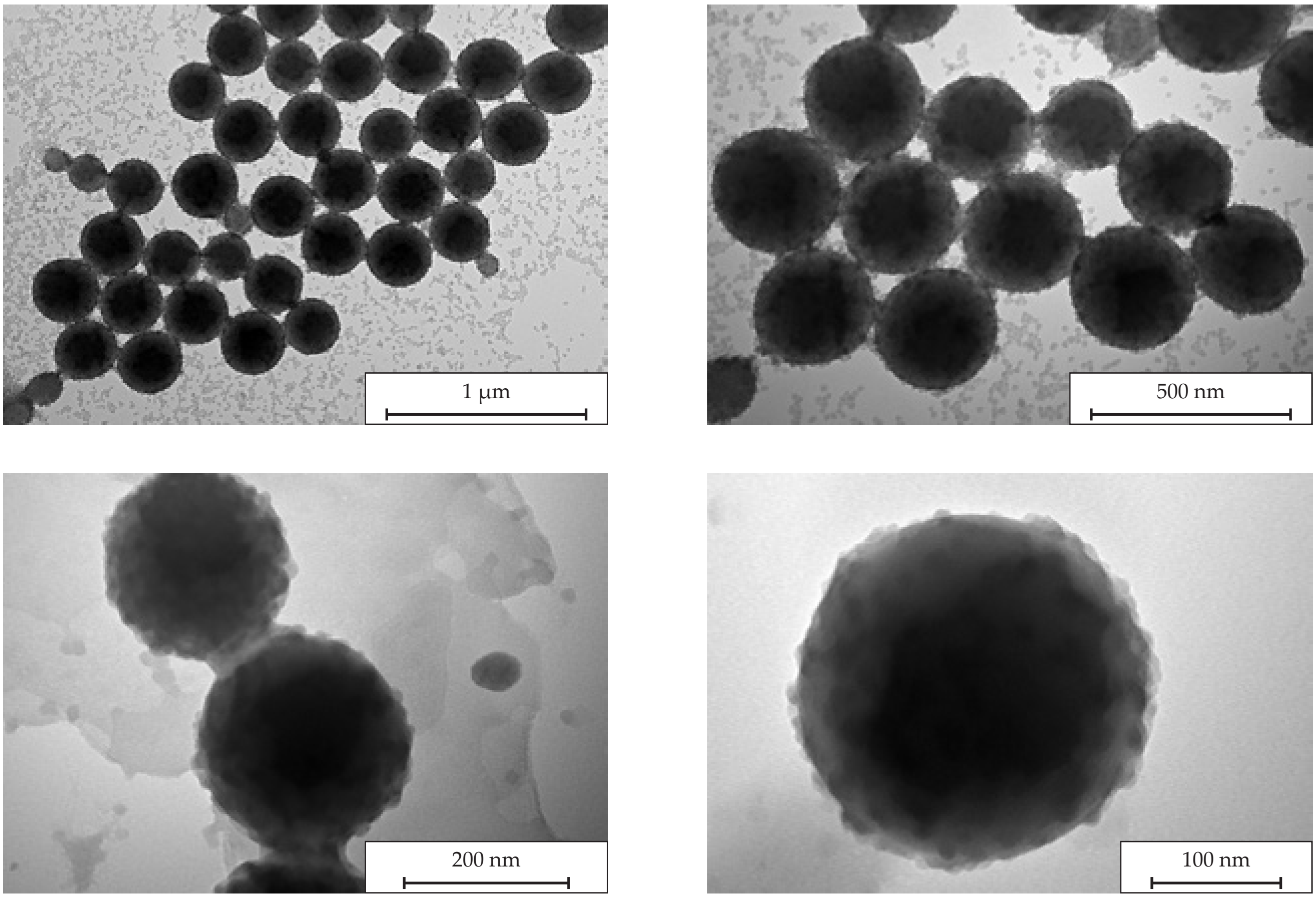

Fig. 5. TEM images of polystyrene latex with monomodal particle size distribution polymerized in 80°C, pH=5, 30% SiO_2/styrene

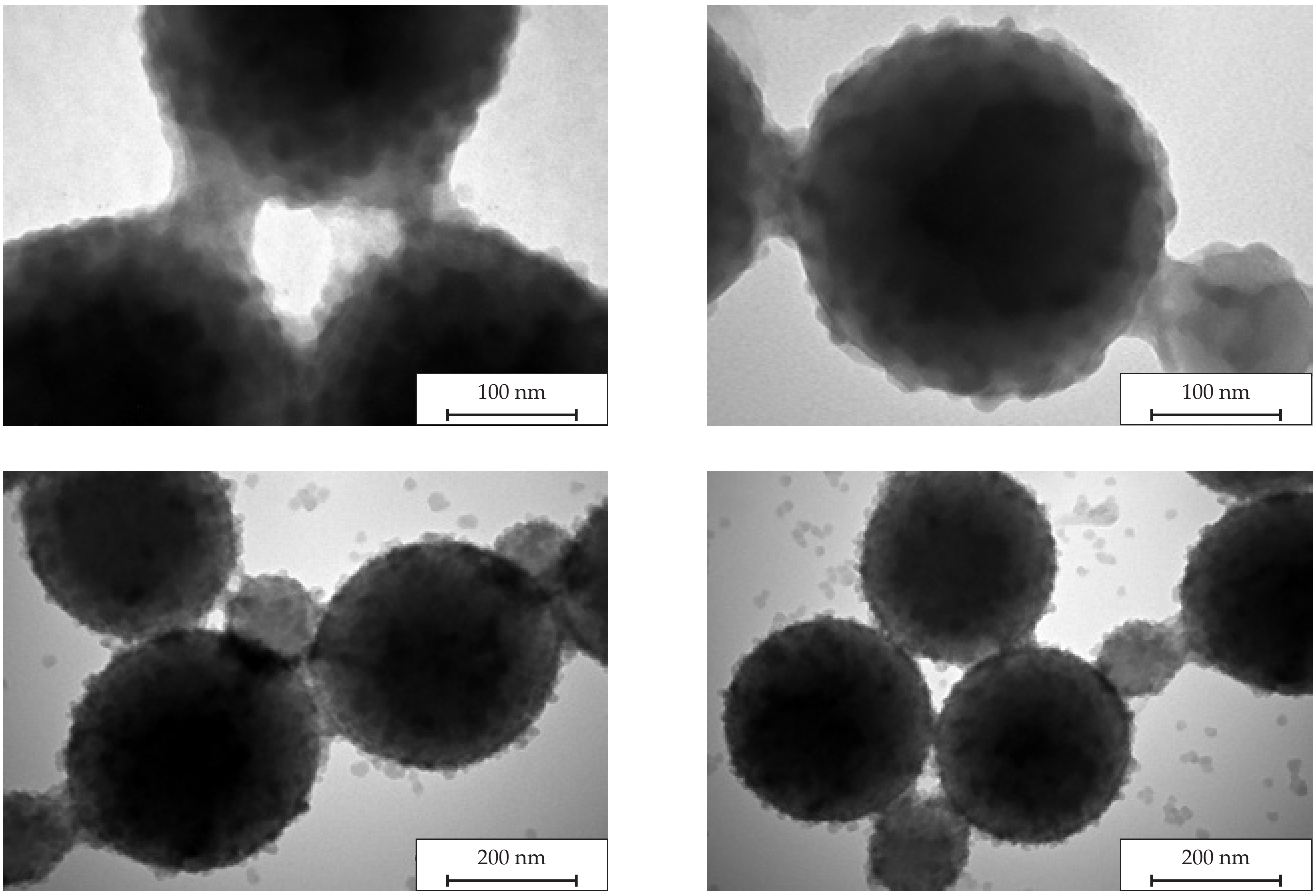

Fig. 6. TEM images of polystyrene latex with monomodal particle size distribution polymerized in 80°C, pH=5, 30% SiO_2/styrene

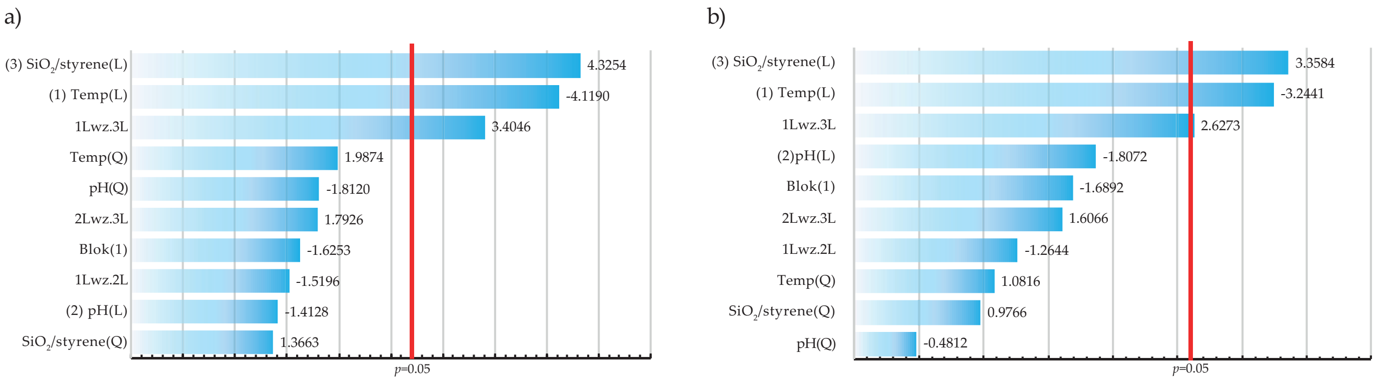

Fig. 7. Pareto chart for: a) dry content, b) conversion observations after 6 hours

erization. In addition, the further split of the peak into a trimodal system was observed because of polymerization at pH 7 in the presence of 20% colloidal silica per monomer charge. The increase in average particle size and the cleavage of the particle distribution because of probable aggregation of polymeric particles may indicate partial release of the initiator from its salt and significant repulsion of negatively charged colloidal silica particles from each other. As a result, there may be inferior coverage by colloidal silica particles of the forming polymer particle. In addition, the separation of the initiator from its salt may contribute to initiation in the oil phase, or to less efficient alpha-functionalization of the forming polymer chain leading to lower affinity of the polymer particle for colloidal silica.

TGA analysis

The solid residue of Pickering emulsion polymerized latex samples was evaluated by thermogravimetric analysis (TGA) after heating to 1000°C. The moisture from the samples was removed at 100°C. The leftover monomers evaporate at 350°C, causing a 2% mass loss. At 600°C, most organic materials pyrolyzed. Between 600 and 700°C, mass decreased slightly. Depending on the colloidal silica amount, the residual ranged from 20-30%. TGA analysis showed that the material remains unaltered at 1000°C. This shows the nanocomposite consists of the proper amount of colloidal silica.

TEM image of polystyrene–silica nanocomposite

This study examined a monomodal polymerization test subgroup. A transmission electron microscope (TEM) imaged the material at various magnifications.

Figure 5a and 5b show most particles with similar dimensions and a minority with smaller diameters. Colloidal silica-coated polymer particles bridged, and a lot of colloidal silica was spread in the continuous phase. Figs. 5b and 5c show that most bridge structures mostly consist of silica nanoparticles. After a few weeks, macroscopic latexes tend to sedimentation and redispersion after agitation. When latex was powdered and mixed in deionized water, it behaved similarly with different degrees of redispersibility. Bridging by silica nanoparticles can be related to this phenomenon as agglomeration and sedimentation are in this case for certain degree are reversible processes which could come from weak interaction forces between colloidal silica particles. Redispersion may be possible if stiff polystyrene particles are segregated by silica nanoparticles. Fig. 5d shows that AAPH-induced silica nanoparticle adsorption onto polystyrene latex extensively coats polymer particles. The dissimilarities in attraction forces between polystyrene and silica nanoparticles, as well as between silica nanoparticles themselves, which lead to their agglomeration onto nanocomposite particles, may reduce stability in a liquid state and cause redispersibility after water mixing. Fig. 6a shows that agglomerated silica nanoparticles likely help the polymer particles bridge. Figs. 6b-d demonstrate unusual sphere microstructures with grey colours like silica nanoparticles. Silica nanoparticles may dominate these microstructures. Furthermore, these bright grey nanoaggregates with a diameter of 80 to 100 nm could be also silica-rich polymer seed produced in the late stage of the emulsion polymerization covered by aggregated colloidal silica induced by AAPH addition to the water phase. Fig. 6c-d shows also colloidal silica agglomeration without polymer particles which can come from the beginning of the process when AAPH was added to the water solution.

Using TEM microscopy to image and evaluate colloidal silica particle size after AAPH addition prior to polymerization may show that the cationic initiator destabilises silica particles. This action may cause silica nanoparticles to agglomerate and adhere to polymer particles. The non-selective destabilisation process may cause sedimentation and redispersion of stiff polymers and silica nanoparticles. Polymers with low glass transition temperatures, below room temperature, may behave similarly to a certain extent. The development of films during polymer mass sedimentation may make redispersion more difficult over time. Colloidal silica may also affect dried polymer film properties. Thus, polymers with a glass transition temperature that allows film formation at ambient circumstances should be investigated further.

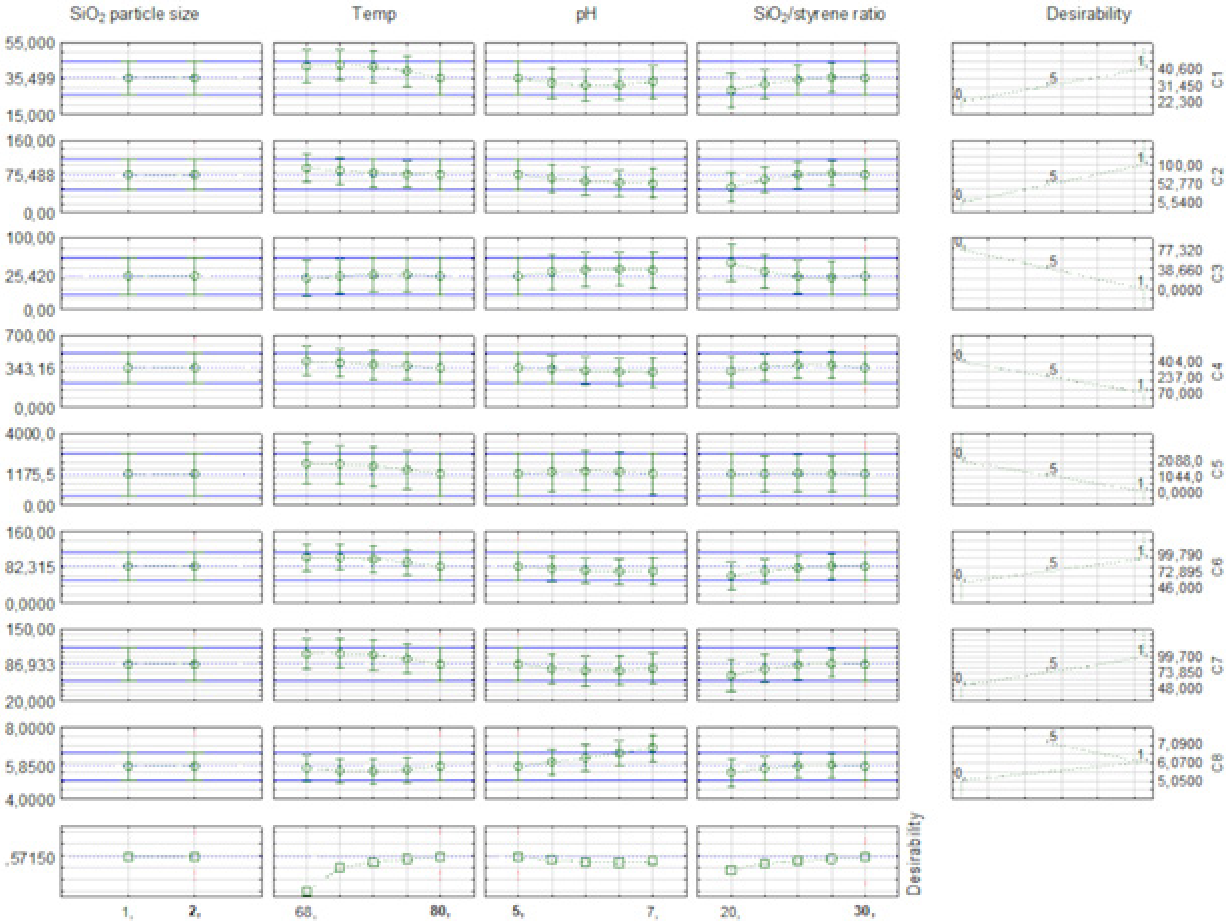

Fig. 8. Profiles of predicted values for best process performance

Data analysis and optimisation

ANOVA analysis shows a substantial positive association between the SiO_2/styrene ratio and the latex's dry matter %. A negative inverse association with temperature and a positive linear interaction effect were also detected. Figure 7a shows that these effects are statistically significant. Figure 7b shows conversion observations after 6 hours.

The statistical study shows that the SiO_2 to styrene ratio increases particle content in the first fraction of the latex. pH negatively impacts the same particles. The temperature-pH negative linear association is noteworthy. Temperature does not significantly affect this parameter ($p>0.05$). The results show that SiO_2/styrene ratio and temperature had a statistically significant positive and negative effect on latex production % conversion after 24 hours. Comparing 24- and 6-hour percentage conversions showed no significant interaction impact. The ratio of SiO_2 to styrene and pH linearly interact to increase the particle content of the second fraction in the final latex. SiO_2/styrene ratio alone negatively affects this parameter statistically. Like the first group of particles, the pH variable interacting with another variable had no statistically significant effect on the particle composition of the final latex ($p>0.05$). The final parameter where independent variables have a statistically significant effect is the latex pH. This study found only positive outcomes for initial pH values. Temperature, initial pH value, and SiO_2/styrene ratio also interact linearly.

Optimalisation of Pickering emulsion polymerisation

A desirability function is needed for a Design of Experiments (DOE) study. All relevant quality variables are weighted in this function to determine product quality. In other words, the comprehensive function of desirability is a weighted mean of several utility functions with user-specified weights. The following requirements were assumed for optimisation. To enhance the clarity of information in the figures, the examined criteria have been designated as C1 to C8.

- percentage of dry content – optimum value 40% (C1)
- percentage of the first fraction – the higher the better (C2)
- percentage of the second fraction – the lower the better (C3)
- average particle size values of the first fraction – the smaller the better (C4)
- average particle size values of the second fraction – the smaller the better (C5)
- percentage of conversion after 6 h – the higher the better (C6)

– percentage of conversion after 24 h – the higher the better (C7)

– pH of final latex – optimum value 6 (C8)

Based on this assumption calculated optimal conditions and the extent to which the assumed parameters for the process under investigation can be achieved.

Optimisation confirmed ANOVA results. Fig. 8 shows that process temperature usually degrades product quality, while the SiO_2/styrene ratio usually improves it. The Pickering emulsion polymerization process can be optimised to produce a product that meets 60% of the assumed criteria. Different parameters' sensitivity to process factors causes this. For instance, while an increase in the SiO_2/styrene ratio increases the proportion of dry content to the targeted 40%, it may only increase the second fraction to 25%. However, it should be noted that, the current situation comprises broad criteria. The lack of a target criterion's minimum or maximum value does not mean that all quality parameters have been met for the product's intended use.

CONCLUSIONS

Polystyrene latexes with different colloidal silica contents were obtained. Decreasing the content of colloidal silica increased the average particles obtained by emulsion polymerization. The increase of the size of the average particles took place with an increase in the modality of the particle size distribution, obtaining bimodal and multimodal distributions. Amount of colloidal silica incorporated into nanocomposite confirmed by thermogravimetric analyses. In addition to the amount of colloidal silica, process conditions were a key factor affecting particle size and distribution. The technique at 80°C in a slightly acidic pH environment produced a monomodal particle dispersion and rapid monomer conversion. However, because stabiliser amount impacts polymerization rate, process temperature should be adjusted to colloidal silica amount. When less colloidal silica is introduced to polymerization, the initiator may burn out before the reaction is complete, therefore lower the temperature may be needed. The choice of initiator is also a key factor influencing the degree of coverage of polymer particles with colloidal silica. In the presented paper, AAPH was used as a radical initiator in the form of a cationic salt. The cationic salt of the initiator destabilises anionic functionalized colloidal silica, increasing its size before polymerization. The destabilisation decreases colloidal silica particle repulsive forces, causing silica to pack densely on the polymer surface. The method may yield poorer results at neutral pH due to the partial release of the initiator from its cationic form and more repulsive interactions between the negatively charged colloidal silica. In addition, the polymerization conducted by Pickering emulsion resulted in latexes characterized by different sedimentation rates, which are reversible. TEM imaging revealed colloidal silica bridges between polymer particles. Bridging may produce sedimentation and liquid redispersion after mixing. Process kinetics are also similar for Pickering and conventional emulsion polymerization. In conclusion, Pickering emulsion polymerization may yield fascinating nanocomposites with unique properties. Colloidal silica as a stabiliser needs additional research to determine sedimentation stability. Water uptake, structural integrity following water immersion, and blocking resistance of polymer films are fascinating.

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- Nowe zastosowania oraz metody badań i właściwości polimerów

Ważne terminy:
Zgłoszenie udziału w konferencji – **30.04.2023 r.**
Termin załączania abstraktów – **15.05.2023 r.**
Termin wnoszenia opłat do – **15.06.2023 r.**

Opłaty konferencyjna:
1750 zł (pełna opłata); 1500 zł (opłata za doktoranta/studenta)
1300 zł (opłata za osobę towarzyszącą)
Opłata konferencyjna obejmuje: uczestnictwo w konferencji, pełne wyżywienie – od kolacji 11 września do obiadu 14 września (śniadania wyłącznie dla gości zakwaterowanych w hotelu Green Mountain), uroczystą kolację, przerwy kawowe, publikację artykułu w pracy zbiorowej: „Modyfikacja Polimerów Stan i Perspektywy w roku 2023", materiały konferencyjne.

Opłata za hotel: opłata konferencyjna nie zawiera noclegów. Każdy z uczestników dokonuje rezerwacji indywidualnie. Cena noclegu dla uczestników konferencji (na hasło MODPOL23) wynosi odpowiednio: 360 zł (opłata za 1 noc w pokoju 1-osobowym), 400 zł (opłata za 1 noc za pokój 2-osobowy – 200 zł/os.).

Miejsce konferencji: Hotel Green Mountain, Karpacz
Szczegółowe informacje już wkrótce na stronie internetowej katedry.

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