

Self-Driving Laboratories for Photopolymer Formulation: Closed-Loop Experimentation, Measurement Quality and Decision Governance

A state-resolved qualification framework for autonomous formulation research

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Abstract

Self-driving laboratories combine automated experimentation with algorithmic experiment selection, but the evidence base is dominated by chemistry and materials platforms outside photopolymer formulation. This review therefore does not assume that general autonomous-laboratory success transfers directly to photocurable systems. It defines the additional state variables required when formulation composition, optical absorption, inhibition, rheology, cure history, washing and post-cure jointly determine the measured outcome. The central engineering proposition is that an autonomous loop is only as credible as its measurement traceability, state definition, feasible-domain controls and decision governance. A closed loop that optimises a convenient proxy while losing formulation identity, specimen history or measurement uncertainty can converge reproducibly on the wrong engineering decision. The review separates exploration, optimisation and qualification; specifies a minimum state record for photopolymer experiments; and proposes stop, escalation and human-review gates for autonomous campaigns.

Keywords: self-driving laboratory · photopolymer formulation · closed-loop experimentation · measurement traceability · autonomous materials discovery · qualification · decision governance

1. Qualification problem and evidence boundary

Self-driving laboratories integrate automated experimental execution with data-driven experiment selection [1-3]. Their value lies in reducing the number of experiments required to map or optimise a design space, not in making evidence independent of experimental quality. The strongest published demonstrations span chemical synthesis, inorganic materials, scattering and other materials-discovery settings [1-4]. Direct evidence for fully autonomous photopolymer-formulation laboratories remains comparatively sparse. Consequently, the transfer from general SDL practice to photopolymer engineering must be treated as a qualification problem rather than as an established performance claim. Photocurable formulations create a particularly demanding closed loop because the measured response depends on both formulation state and manufacturing history. Composition, initiator and absorber levels, inhibitor state, viscosity, dissolved oxygen, filler dispersion, spectral irradiance, dose, temperature, layer geometry, washing and post-cure can all alter the observed output (see ER-035, ER-351, ER-352 and ER-359).

The autonomous controller must therefore operate on a declared state vector rather than on an anonymous recipe identifier.

2. What the loop is allowed to learn

Three tasks must be separated. Exploration seeks information about the response surface; optimisation seeks candidates that improve an objective under constraints; qualification determines whether evidence is adequate for a defined use. Conflating these tasks creates circular reasoning: the same adaptive data used to discover a candidate cannot automatically be treated as independent qualification evidence. A valid loop should distinguish controllable formulation variables, controllable process variables, nuisance variables and measured responses. Lot identity, instrument calibration, environmental conditions and specimen age belong to the state record even when they are not optimisation variables. Variables that cannot be measured reliably should not be silently absorbed into model error.

3. Photopolymer-specific state architecture

The minimum formulation state should include chemical-family identity, component fractions or bounded coded variables, solids fraction where relevant, mixing sequence, mixing time, elapsed time since preparation, temperature and any controlled atmosphere. The optical-process state should record wavelength or spectrum, irradiance measurement geometry, exposure duration or dose, layer thickness and the presence of attenuation or scattering species. The post-exposure state should record the interval to measurement, wash conditions, drying and post-cure. This architecture is intentionally technology-neutral. Radical, cationic, anionic, hybrid and step-growth photocurable systems can differ in inhibition, dark reaction, conversion history and post-exposure evolution. A controller that assumes one common response law across such regimes risks creating a statistically smooth but chemically invalid search space.

Table 1. Minimum state vector for an autonomous photopolymer experiment

Domain	Required record	Failure if omitted
Formulation	component identity; coded fraction; lot; mixing sequence; age	composition drift or untraceable candidate
Optical process	spectrum/wavelength; irradiance; exposure/dose; geometry	non-transferable cure response
Thermal/environment	temperature; atmosphere/oxygen; humidity where relevant	hidden kinetic or inhibition shift
Post-processing	wash; dry; post-cure; elapsed time	measurement state not comparable

Domain	Required record	Failure if omitted
Measurement	method; calibration; replicate; uncertainty; raw data pointer	optimiser learns instrument bias

4. Measurement quality is part of the control law

Autonomous formulation platforms can prepare reproducible mixtures, but published liquid-handling and formulation systems also show that dispensing geometry, physical setup and measurement integration matter [4]. A photopolymer loop should use gravimetric or otherwise traceable verification where feasible and should retain raw measurement data, not only transformed scores. Each response used by the controller needs a measurement model: measurand, unit, method, calibration state, replicate structure, uncertainty estimate and failure criteria. A cure-depth value, viscosity value, tensile modulus and optical transmittance are different evidence objects. Combining them into a scalar objective is a decision operation and should remain reversible to the underlying measurements.

5. Candidate selection and feasible-domain governance

Candidate selection must be constrained by chemistry, equipment and safety. Hard constraints exclude mixtures that cannot be dispensed, mixed, illuminated or handled within the authorised laboratory envelope. Soft constraints encode regions that may be feasible but have poor expected manufacturability. The feasible domain should be versioned because equipment changes, raw-material substitutions or improved handling may alter it. The controller should never extrapolate a candidate into an undeclared region merely because a surrogate predicts improvement. Out-of-domain proposals should trigger an explicit escalation path: reject, request human review, or run a bounded diagnostic experiment designed to test feasibility rather than performance.

6. Replication, randomisation and drift

Adaptive campaigns are vulnerable to time-correlated drift because experiments are not sampled independently of sequence. Resin ageing, ambient temperature, lamp drift, tip wear, balance drift and contamination can become spuriously associated with candidate composition. Replication and periodic reference formulations are therefore control elements rather than optional statistical refinements. At least one stable reference condition should recur across a campaign at a frequency justified by expected drift. Randomisation or blocked sequencing should be used where compatible with the process. A controller should be able to distinguish a changed material response from a changed measurement system.

Table 2. Autonomous-loop gates

Gate	Question	Action on failure
Feasibility	Can the candidate be prepared and processed safely?	reject or human review
Measurement validity	Is the response traceable and within method capability?	repeat or quarantine
Drift control	Does the reference remain in control?	pause campaign and investigate
Model domain	Is the proposal supported by nearby evidence?	diagnostic experiment or reject
Qualification	Has the final candidate passed independent confirmation?	no release claim

7. Decision governance and authority

Safe SDL operation requires explicit authority boundaries [3]. In photopolymer development, the automation may be authorised to select experiments within a bounded formulation and process domain, but changes to hazardous-material classes, thermal limits, irradiation hardware, waste handling or human exposure conditions require separate approval. Decision logs should capture the candidate proposed, model version, training data snapshot, constraints applied, predicted objective, predicted uncertainty, reason for selection and final execution status. This allows later reconstruction of why an experiment occurred and prevents a final optimum from being detached from the path that created it.

8. Qualification tests that must remain independent

An autonomous optimisation campaign can identify promising candidates, but release-grade claims require independent confirmation under locked methods. Confirmation should use fresh preparation, predefined acceptance criteria, appropriate replication and measurement methods that were not changed to fit the candidate. If the intended use depends on service ageing, sterilisation, biocompatibility, fire, electrical or other application-specific properties, those domains require their own qualification evidence. The key boundary is non-compensatory: a high optimisation score cannot compensate for a failed safety, stability, reproducibility or measurement-validity gate.

9. Minimum reporting record

A publishable autonomous-formulation study should report the searchable variable space, fixed variables, constraints, experimental executor, measurement methods, uncertainty treatment, surrogate or selection method, acquisition rule, initial design, stopping rule, failed experiments, rejected candidates, human interventions and independent confirmation. Without this record, apparent acceleration cannot be separated from hidden manual correction. For photopolymer studies, the publication should additionally state spectrum or wavelength, irradiance or dose definition, sample geometry, oxygen/environment control, thermal state, wash and post-cure history and elapsed time to measurement.

10. Qualification decision boundary

Self-driving laboratories can make photopolymer formulation research more efficient only when automation preserves the state information required for causal interpretation and later qualification. The appropriate target is not an unattended optimiser. It is a traceable experimental system in which every autonomous action remains bounded by measurement quality, feasible-domain controls, reproducibility checks and human authority. The evidence reviewed supports autonomous experimentation as a platform concept, but not

a universal claim that autonomous formulation produces superior photopolymers. That conclusion remains material-, workflow- and endpoint-specific.

11. Experiments that discriminate a useful loop from a fast loop

A credible qualification campaign should deliberately perturb the elements that an autonomous system might otherwise hide. One test should hold formulation constant while changing measurement order to reveal temporal drift. A second should repeat a reference formulation across the full campaign to estimate combined executor and analytical stability. A third should introduce blinded replicate preparations so that the controller cannot exploit sample identity or sequence. A fourth should rerun selected candidates with a fresh preparation and independently reset instruments. Agreement across these tests provides evidence that the loop is learning material response rather than campaign chronology. An additional challenge experiment should place a candidate near a known feasibility boundary, such as high viscosity, strong attenuation or marginal cure. The desired outcome is not necessarily successful manufacture. The purpose is to verify that the system detects reduced measurement validity or elevated uncertainty and escalates rather than converting a borderline execution into a high-confidence data point.

12. Failure signatures and false diagnoses

A self-driving formulation campaign can fail while every individual software component appears functional. Repeated improvement in the objective accompanied by progressive drift in a reference sample indicates that the optimiser may be following instrument change. Strong dependence of selected composition on experimental order suggests nonstationarity. Frequent human rescue of the same proposal class indicates that the formal feasibility model is incomplete. A large gap between automated and independently prepared confirmation samples indicates executor or mixing bias rather than model error alone. False diagnosis is especially likely when a convenient proxy is treated as the target. A formulation may show increased cure depth because optical attenuation has decreased while conversion at the required geometry remains inadequate (see ER-035 and ER-352); viscosity may appear acceptable at one shear condition while recoating fails; a mechanical endpoint may improve after a post-cure change rather than because of the selected formulation. The loop must retain the causal path between controls, intermediate states and final objective.

13. Transfer limits and requalification

Transfer should be evaluated as a change in state distribution. Moving from one photopolymerisation wavelength to another, changing irradiance geometry, replacing a balance or dispenser, changing vessel scale, introducing a new raw-material lot policy or altering post-cure can all change the mapping learned by the loop. A previous model can be used as prior information, but it should not silently become the controlling model for the new domain. Requalification depth should reflect the change. Minor executor maintenance may require reference-material checks and metrological confirmation; a new chemistry family may require a new search-space definition, safety assessment and initial design. The governing principle is that autonomous history is reusable only to the extent that state equivalence is demonstrated.

14. Evidence gaps and research priorities

The most important evidence gap is the limited number of published, fully traceable photopolymer SDL studies that connect autonomous formulation selection to state-resolved cure, printing and post-processing outcomes. Future work should report negative experiments, executor failures and human interventions rather than only the final optimum. Benchmark problems should include discrete chemistry choices, process-dependent feasibility and at least one deliberately drifting measurement channel. Cross-laboratory replication is also needed. A useful benchmark would distribute the same bounded formulation problem, reference materials and reporting schema to independent laboratories using different executors. The objective would be to test whether autonomous decisions converge after accounting for measurement uncertainty, not whether identical recipes are selected under non-equivalent hardware.

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Competing interests

The author is Founder, CEO and CTO of 3Dresyns by Resyner Technologies S.L.; ER-401 contains no comparative recommendation between commercial products.

Data availability

ER-401 is a literature-based engineering review; no new experimental dataset was generated.